COKE AND BY-PRODUCTS MANUFACTURE

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PREFACE

It was suggested to me that my lectures on Coke and By-products Manufacture, covering the syllabus of the City and Guilds of London Institute examinations, would form a basis for a greatly needed book on the subject.

I have therefore brought the matter up to date, and it is offered in book form in the hope that students sitting for the examinations of the City and Guilds of London Institute and junior members of the Coke-Oven Managers' Association and of the Institute of Gas Engineers will find what they need herein. It will also show something of the advances made in Great Britain by this industry during the past eighteen years.

The Coke-Oven Managers' Association are proposing to require qualification by examination from applicants for Associate Membership, and from October 1941 such applicant under the age of 35 years shall be required to furnish evidence that he has obtained—a First Class Pass in the Final Grade of the City and Guilds of London Institute examination in the subject of " Coal Processing, Sect. 4a : Coke and By-product Manufacture," or a Science Degree or other qualification. Thus this book, based on the above City and Guilds examinations should be of interest to aspirants to Membership of the Coke-Oven Managers' Association.

I wish to place on record my thanks to the Koppers Coke-Oven Co. Ltd., the Rheolaveur General Construction Co. (1939) Ltd., Simon-Carves Ltd., and the Woodall-Duckham Vertical Retort and Oven Construction Co. (1920)

Ltd., and my appreciation to them for kindly supplying data of modern plant.   I also wish to thank many colleagues for their kindness and help in revising notes.

<div align="right">E. C. DIXON.</div>

BLACKHILL,

  Co. DURHAM.

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INTRODUCTION

HISTORICAL ASPECTS—BEEHIVE TO MODERN REGENERATIVE OVEN

COKE is the solid residue which is produced when coal is heated in the absence of air, or in the presence of only a limited supply, *i.e.* during the destructive distillation of coal. It is chiefly used for the smelting of iron. Iron was used many thousands of years ago, but until the manufacture of steel was developed by Bessemer and Siemens, causing a demand for pig iron and thus coke to smelt it, the manufacture of coke on a large scale had not been modernised. Prior to 1750 to 1780 mainly wood charcoal had been used in the smelting of iron. It was only about that date that the general use of coke began.

The original coke-oven was very primitive, generally a heap of large coal covered with soil or straw around a central brick chimney. A fire was lit at the base of the chimney and the heat gradually penetrated the whole pile ; in ten days or more it was quenched and the coke was drawn. Coke was the only product obtained. A semi-combustion process took place resulting in the loss of the whole of the gas and by-products together with part of the coke, probably 15 to 20 per cent. of the coke being burned.

From then up to about 1850 coking between walls was employed, in which the amount of air needed for partial

burning could be better controlled than in open piles. The beehive oven was later made more efficient by building the ovens in double rows with a central waste heat flue between, through which the hot gases were drawn for steam-raising purposes. Thus, gradually, the beehive oven was adopted for the extraction of by-products. In 1877 there were 14,000 beehive ovens in operation in the Durham coalfields, producing some four million tons of coke.

In 1862 the Carve Company built a battery of Knab ovens, wherein the gas, after condensation of tar and liquor, was used to heat the ovens at the bottom and also side flues. Later they reduced the width of the Carve oven to 24 ins. and the coking time to 24 hours. In 1882 the first battery of Simon-Carve coke-ovens erected in this country was built for Messrs. Pease & Partners at Crook in the county of Durham. The ovens were 23 ft. long, 6 ft. 6 ins. high and $19\frac{1}{2}$ ins. wide. They held $4\frac{1}{4}$ tons of coal, coking in 60 to 72 hours. They were in use until recently.

Coppée in Belgium about 1861 introduced a non-recovery oven, with a narrow chamber of 18 ins. and vertical flues, $29\frac{1}{2}$ ft. long and $3\frac{1}{2}$ ft. high, with a charge of 3 tons, coking in 24 hours. The Otto Company introduced this oven into Germany in 1867, and in 1881 adapted it for the recovery of by-products.

Hoffman, in collaboration with Otto, built the first Otto-Hoffman regenerative oven in 1883 with twenty-eight vertical flues, divided into two sections, united at top and bottom by horizontal flues. Regenerators were used for both gas and air, and the direction of the gas flow was altered periodically.

The Otto-Hilgenstock oven was introduced in 1896. It was without regenerators and was heated by eight burners on the Bunsen principle, the burning gases passing up vertical flues to a top horizontal flue and descending by vertical flues at one end to the main waste-gas flue. In 1900 the number of burners was increased to ten, enabling

a 7½-ton charge to be carbonised in 30 hours. In 1905 the Otto oven was heated by twelve burners, and in 1910 this was increased to sixteen, so that one burner supplied gas to two vertical flues. In 1914 the Otto oven was one of the most popular in this country. They were all built with semi-silica material.

In America, however, about this time they began to experiment with ovens built with silica material, which enabled higher temperatures to be used and much more rapid coking times. In England little change took place, although many firms had experimented with a few silica oven-walls, until the year 1924 when the Consett Iron Company started up a battery of sixty silica ovens. This battery at Consett was the first modern battery of American-type silica ovens erected in this country complete with modern equipment such as electric coke-quenching car and coal-loading car, and other mechanical labour-saving devices. It was built by the Coppée Company (Great Britain) Ltd. The sixty Wilputte ovens were built of high-grade silica shapes made at Consett. The ovens were of regenerative type, 40 ft. long, 10 ft. 6 ins. high and 17¾ ins. mean width, coking 11½ tons of coal in 15½ hours. Surplus gas was supplied to the adjoining steelworks.

Thus there began in this country a period of somewhat slow but steady rebuilding and modernising of coke and by-products plants. Silica material and more rapid coking with consequent cheaper labour costs became universal in the new batteries. Higher ovens with charges up to 18 tons were built. Whether there is no limit to size and speed or whether if carried too far the product will suffer, the future will tell.

The first battery to be heated with blast-furnace gas was the Becker ovens of Messrs. Lysaght Ltd., Scunthorpe, Lincs., in 1932 ; thus all the gas generated was available for other uses.

These modern ovens are generally top-charged, have a

regular operation and improved heating flues, and the coke, though smaller than beehive coke, is much more uniform in quality. The faster rate of heating also gives higher benzole yields. Modern silica ovens with a charge of up to 18 tons, coking in, say, 16 to 18 hours and with a through-put of, say, 26 tons of coal per oven daily, as compared with the daily throughput of the older types of 5 to 6 tons, show remarkable improvement. Thus the modern oven, with a greater coking capacity than that of the oven considered suitable under pre-war conditions, requires a much smaller number of ovens to coke a given quantity of coal daily.

The increase in regenerative ovens has made the amount of surplus gas available for sale, either for town's use or for use in steelworks, etc., greater and greater, until gas has become one of the most valuable by-products. On the other hand, the decrease in price received for sulphate of ammonia has made that by-product barely profitable to make, which is partly due to its synthetic manufacture from nitrogen and hydrogen in large quantities. Against this the Sulphate of Ammonia Federation has greatly improved this product and increased its sale, although its price may be only half that received some twenty odd years ago.

In pre-war days not every coke-oven plant made benzole ; now it is an important by-product. The modern oven has increased the benzole yield. The quality of motor benzole has been standardised and greatly improved through the National Benzole Association. Crude tar has been improved and standard road tar is a valuable product. Other by-products regularly manufactured are pure benzene, xylene, toluene, solvent naphtha, carbon bisulphide, creosote, anthracene, naphthalene and pitch. Nowadays coke itself is not produced only for blast-furnace or foundry use, but a growing market is developing for it as a smokeless fuel for household purposes. Improved organisation, combined with research and study, may show that the future holds further improvement and development for the coking and

by-product industry. Probably more uses will be found for coke and its many by-products, and the coke-oven manager will have to be a man of vision to expand the industry in the future as it has expanded in the past decade.

CHAPTER I

COMPOSITION AND PROPERTIES OF COALS

COAL is a mineralised vegetable matter, being a rock-like portion of the earth's crust, derived from plant life which has undergone change until it consists almost entirely of its principle component carbon. Lewis describes coal as an agglomeration of the solid degradation products of vegetable decay, together with such original bodies as have resisted to a great extent the action to which the material has been subjected.

Coals may be classified as :—

(1) *Sub-bituminous* coals with 40–50 per cent. volatile matter, 55–60 per cent. fixed carbon, 15–20 per cent. oxygen and nitrogen ; residue non-coherent, high-volatile and non-coking.

(2) *Bituminous* coking coals can be divided into a further three groups :—

    (a) 32–40 per cent. volatile matter, 60–68 per cent. fixed carbon, 10–14 per cent. oxygen and nitrogen ; residue porous open coke. This is high-volatile coking coal.

    (b) 26–32 per cent. volatile matter, 68–74 per cent. fixed carbon, 5·5–11 per cent. oxygen and nitrogen ; residue hard porous coke. This is normal coking coal.

    (c) 18–26 per cent. volatile matter, 74–82 per cent. fixed carbon, 5·5–6·5 per cent. oxygen and nitrogen ; residue very dense coke. This is low-volatile coking coal.

(3) *Semi-bituminous*, steam coals with 15–20 per cent. volatile matter, 80–85 per cent. fixed carbon, 4–4·5 per cent. oxygen and nitrogen; residue is non-coherent and non-swelling.

(4) *Anthracite*—smokeless steam coals with 8–15 per cent. volatile matter, 85–92 per cent. fixed carbon, 2–4·5 per cent. oxygen and nitrogen; residue non-coherent.

(5) *Anthracite* coals with volatile matter below 8 per cent., fixed carbon above 92 per cent., oxygen and nitrogen 2–4·5 per cent.; residue non-coherent.

The Evaluation of Coals.—Coals may be evaluated for :—

(1) Coke and by-products. This may be carried out by coking a weighed sample of coal and weighing the products.

(2) Gas production.

(3) Steam-raising.

(4) Hydrogenation.

(5) Household and other purposes.

Composition

(1) Ultimate chemical analysis of coal—hydrogen, oxygen, nitrogen, carbon and sulphur.

(2) Proximate analysis—ash, volatile matter, fixed carbon, etc.

Constitution

(1) Microscopic examination.

(2) Action of solvents.

(3) X-Ray examination.

Dr. Stopes and Professor Wheeler have examined coal

microscopically and divided it into the four main types :—
fusain, durain, clarain and vitrain.

Fusain is a dull black, soft, crumbly substance existing
in thin layers, of a non-coking nature, very dusty and
assumed to be the cause of initial spontaneous combustion.
It has a high ash content and the same features as powdery
anthracite, is absorbent and collects salts—hence the ash
is often highly coloured by iron compounds.

Durain is a dull, hard material having a fracture like
pitch. It is lustreless or dull, containing little moisture and
a large percentage of ash. It is very weakly coking, but
may have vitrain bands improving its coking quality. It
is usually found in steam and household coals.

Clarain is extremely bright with a shell-like structure,
rich in plant tissues. It is glossy in appearance and trans-
parent in thin sections. It is brighter than durain but
duller than vitrain. It is suitable for gas coals and occurs
in bands $\frac{1}{8}$ to $\frac{1}{4}$ inch thick, which may be associated with
bands of durain.

Vitrain is bright and glossy with no cleavage planes. It
is found in smaller quantities in cube-like formation, bright
and brilliant in appearance. It is usually seen in soft coal ;
unlike clarain, it is devoid of plant structure. One feature
which distinguishes vitrain from clarain is that, if fusain
is found upon its surface or in contact with it, after removing
the fusain there is no imprint of it on any part of the vitrain,
whereas the imprint of fusain is clearly seen upon clarain.
Vitrain enhances the coking properties of fusain. Generally
speaking, vitrain yields the most gas and the least tar.

The proportion of volatile matter and gas increases slightly
in passing along the series durain—clarain—vitrain. While
clarain and vitrain evolve more gas than durain, the rates
of production are the same.

Alpha, Beta and Gamma Compounds.—In the resolution
of coal by means of solvents, phenol or pyridine is first
added to dissolve out the *beta* and *gamma* compounds,

leaving behind an insoluble residue of a so-called *alpha* compound. The *beta* and *gamma* compounds are then separated by chloroform. The outstanding points are that the *gamma* compound has a very high hydrogen content, is high-volatile, and that it melts, giving rise to liquid by-products of low phenolic content. The *beta* compound has a high oxygen content due, no doubt, to the presence of a cellulose residue wherein the oxygen is associated with hydrogen in hydroxyl, carboxyl or pseudo-acid groups. This type is devoid of coking quality. It gives rise to highly phenolic by-products and a relatively weak gas.

The hydrogen is some measure of the coking or cementing properties of a coal under the influence of heat. The *gamma* compound has a high hydrogen content associated with very little oxygen, so it has been stated that a high hydrogen associated with a low oxygen content is a criterion of coking quality.

Impurities in Coal.—The impurities in coal are of great importance in coking. The chief impurities are ash, sulphur, phosphorus and chlorine (chiefly in the form of sodium chloride, common salt).

Ash is composed of the incombustible mineral matter left as a residue on completely burning coal, coke, etc. It is variable in composition, consisting chiefly of silica, 30–60 per cent., alumina and oxide of iron, 5–10 per cent., along with smaller quantities of magnesia and alkalies. Some of these substances are combined with sulphuric acid, 3–8 per cent., as sulphates.

An excessive ash content is objectionable. In fuels for steam-raising it reduces the calorific value. In order to attain fuel economy in the manufacture of pig iron, much attention has been given lately to the cleanliness of coke and coal. This problem finds its origin in the mining of the raw coal. Owing to a number of narrower seams being

worked, and to the increased use of mechanical coal cutters, there has been a tendency for coal brought to the surface to contain more ash.

Coal has two kinds of ash :—

(1) Inherent or combined ash.
(2) Free or extraneous ash.

The Combined Ash may be defined as that portion of the inorganic constituents which is inseparable from the coal after the latter has been crushed to approximately 80 per cent. through $\frac{1}{8}$-inch mesh. In most coal-seams greater or lesser quantities of ash are found to be intimately associated with the coal substance in such a way as to be inseparable until the coal has been crushed to a considerable degree. In estimating this inherent ash it is usual to crush the coal to a fineness of $\frac{1}{8}$ inch and then subject it to a series of float and sink tests in order to separate the purest coal. The amount of ash found in the lightest portions of the coal is then designated the inherent ash of that coal.

This inherent ash (*a*) is always relatively finely divided ; (*b*) in a given seam the proportion of this ash is nearly constant ; (*c*) when the coal is merely passed over screens and sorting belts at the pithead, the combined ash is still found to be evenly distributed to a greater or lesser degree.

The Free Ash is of considerable importance for the following reasons :—(*a*) it is found in the coal as pieces of inorganic matter variable in shape, size, specific gravity, etc., is derived from bands within the seam, from the roof to the floor, and in consequence varies very often both in chemical and physical properties ; (*b*) it may be either finely divided or otherwise, according to its nature *in situ*, condition of mining, etc. ; (*c*) its percentage in terms of total coal is variable. It has been found in County Durham that the free ash is often of a very definite size and shape, and is therefore roughly separable from the rest of the coal by means of a screening process.

The colliery manager is not always able to control the ash in the coal raised, largely because free ash may exist in smaller pieces than could reasonably be picked up on the sorting belts, and its very presence and nature may be due to geological and other conditions over which he has no control. A variable ash content has had a bad effect on blast-furnace working. It has been stated that a blast-furnace coke with a regularly high ash is more acceptable to the pig iron manufacturers than one whose ash varies from a low figure of, say, 7 per cent. to a high figure of 15–16 per cent., the reason given being that the coke to burden ratio supplied to the blast-furnace must be sufficient to take care of all ash fluctuations, and in consequence a large amount of coke is unnecessarily burned and the quality of the iron produced varies. Roughly speaking, a coal giving 7 per cent. ash will make coke containing 10 per cent. ash. As far as blast-furnaces are concerned, oxide of iron in the ash is not detrimental, but the silica requires additional limestone to flux it.

Iron.—The colour of the ash is a fairly reliable indicator of the proportion of oxide of iron, a deep red colour showing a high content of iron. As the iron compounds spring from sulphide of iron in the coal, a red ash would indicate also a high proportion of sulphur in the coal, this being a most objectionable impurity.

Sulphur exists in coal in four conditions :—

(1) As iron pyrites (coal brasses, iron bisulphide).
(2) As sulphate of lime or alumina.
(3) In an organic form, combined with carbon and hydrogen.
(4) In rare cases as free sulphur.

Of these forms, that which occurs as pyrites is the most injurious, since on combustion of the fuel the sulphur

becomes sulphurous acid and, by further oxidation, sulphuric acid. This has an intensely corrosive effect on any iron or steel with which it may come in contact, especially the cooled surfaces of economiser tubes or other parts on which moisture can condense, as these absorb acid fumes more readily. The sulphur in the form of sulphate is not injurious, except that it adds to the mineral constituents or ash of the coal. Excessive sulphur or phosphorus produces undesirable properties in iron for foundry work especially. In coking practice some of the sulphur passes off with the volatile matter, part remains fixed in the coke, while part is liberated in quenching.

Fixed sulphur left in coke .   .   approx. 70 per cent. of total
Free sulphur in gas   .   .   .   approx. 20 per cent. of total
Free sulphur in liquor and tar   .   approx. 7 per cent. of total

Chlorine is usually found in coal as sodium chloride. This alkali is an important factor in corrosion of oven-walls. In Yorkshire and the Midlands this causes semi-silica oven-walls to require rebuilding in about nine months. Corrosion may be caused by :—

(1) Total soluble salts, not necessarily chlorides.
(2) Deposition of carbon in brick pores.
(3) Volatilisation of iron chlorides with deposition of iron.

This corrosion has caused many ovens to be rebuilt in silica material, which so far as experience shows can stand up very much better to salt corrosion.

Professor Cobb of Leeds gave the following explanation of the action of salt. If coal containing more than 0·05 per cent. sodium chloride is charged into an oven with the walls of semi-silica or fireclay material, these walls suffer. The salt volatilises, penetrates the brick and combines with its constituents. Near the brick face in contact with the coal the temperature is not high enough to cause fusion, but as we approach the face of the brick in contact with

the flue-gases, we arrive at a point at which the sodium silicate and sodium aluminate begin to fuse. This portion becomes porous, honeycombed and weak in structure, setting up disintegration often severe enough to cause the brickwork face to fall away in patches or layers. Pure silica bricks are more resistant as they contain no alumina. The quality of the washing water should not be overlooked, as in many cases such waters contain excessive quantities of sodium chloride, and also sodium sulphate. The chlorine liberated from the salt combines with the ammonia, forming ammonium chloride, which may give rise to thickening of tar in the mains, and this is likely to be aggravated if magnesium chloride is present.

CHAPTER II

COAL CLEANING

Reduction of the Ash Content of Coal.—It is obvious that it is desirable to reduce the ash content of most coals in order that the ash content of the coke for blast-furnace use may be at a reasonably low figure. Gill of Consett stated that a reduction of ash in coke from 12·15 per cent. to 9·8 per cent. resulted in a saving of 1 cwt. of coke per ton of pig iron, and an increased iron output of 15·4 per cent., the quality of the iron being also improved. Further, Mott has analysed the findings of various workers in this field, and finds close agreement between practical results, obtained by Gill at Consett and Declaye in Belgium, and theoretical calculations by Evans at Deladrière, which show that an increase of 1 per cent. of ash in a blast-furnace coke results in an increased consumption of 0·4 cwt. per ton of pig iron. Taking this further and considering the financial value of 1 per cent. of ash reduction to the blast-furnace manager, Mott summarises the findings of six independent workers by saying that a reduction of 1 per cent. of ash in coke is worth at least sixpence per ton of coke to the iron maker. The problem of the coke-maker is whether the ash content of coke can be reduced by 1 per cent. for less than this to allow him a profit.

The run of mine coal ash may be classified as an inorganic residue consisting of a calcined clay substance with varying amounts of other constituents, such as iron oxide, lime, magnesia, titanium oxide, alkalies, sulphuric anhydride, etc., either free or in a state of combination.

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It has been stated by S. Tweedy (*C.O.M.A. Year Book* 1936, 290) that the fusion temperature becomes lower as the size of coal decreases, until a minimum is obtained from the dust, and that the formation of clinker in practice is influenced by many factors other than the fusion temperature of the average ash as determined in the laboratory. Among those he mentioned were :—(a) size of fuel ; (b) amount of ash in fuel ; (c) distribution and properties of the constituents of the ash ; (d) conditions of the burning process, such as rate of burning, length of time the ash is subject to a high temperature and method of firing ; (e) strength of the clinker formed and facilities for its removal. Nichols and Selvi found that the quantity of clinker formed is large for coals of low ash fusion temperature, but decreases as the fusion temperature rises to about 1430° C., and from this point the quantity does not change much. Coke ash should preferably fuse at 1430° C. or higher.

Coal Cleaning processes have been classified by Chapman and Wheeler as under :—

Wet Cleaning Processes.

(1) Those in which a current of water is employed.
      (a) A vertically upward current (Draper washer).
      (b) A current alternately upward and downward (Baum washer).
      (c) A current flowing over an inclined plate (Rheolaveur washer, the Deister-Overstum concentrating table).
(2) Those in which a medium of specific gravity greater than that of water is employed (the Chance, Barvoys and Trompe processes).
(3) Froth flotation processes (the Mineral Separation Co. Ltd. process).

Dry Cleaning Processes.

(1) Those in which a vertically upward current of air is used (Wye separator).

(2) Those in which advantage is taken of the different coefficients of friction of coal and shale (spiral separators).

(3) Separation by centrifugal force.

The objects of coal cleaning are :—

(1) To purify the coal from stone, shale and inorganic matter which would result in a high ash content.

(2) To enable fine coal too small for hand picking to be cleaned.

All wet processes and most dry processes of cleaning coal depend upon a separation by reason of the difference in specific gravity that exists between the coal and the dirt. Pure coal contains no dirt and usually has a specific gravity of less than 1·35 ; whereas dirt, consisting largely of minerals derived from clay, possesses a specific gravity of approximately 2·5, and the value for pyrites is 5·0. Naturally, mixtures of dirt and coal possess specific gravities intermediate between these figures. Herein lies one of the principal difficulties of coal cleaning by any process, because quite a number of coals, forming no inconsiderable fraction, are known as " intermediates," and consist of coal and shale so closely intergrown that very little separation can be secured even by fine crushing. This fraction must go as dirt, if clean coal is desired. Probably it would be best separated as a third product and used for boiler firing, or, when combination ovens are in use, for the producers.

The second difficulty in cleaning coal lies in the relative behaviour of the particles of various sizes.   A particle falls in water with a velocity V given by the equation :

$$V = C\sqrt{d(s-1)},$$

where C is a constant, d is the diameter of the particle, and

s is the specific gravity of the particle. Consequently, $V \propto \sqrt{d}$, and the relative values of V for different values of d are as follows :—

d =	$\frac{1}{200}$	$\frac{1}{120}$	$\frac{1}{64}$	$\frac{1}{32}$	$\frac{1}{8}$	$\frac{1}{4}$	$\frac{1}{2}$	1	2 ins.
V =	0·07	0·09	0·12	0·18	0·35	0·5	0·71	1	1·4

It is thus shown that with the smaller sizes there is very little difference in velocity, and consequently, separation cannot be effected under industrial conditions of working. Industrially there is a limiting size of particle beyond which separation cannot be effected, and this may vary in practice from $\frac{1}{16}$ inch to $\frac{1}{120}$ inch, according to the nature of the coal. Further, it is clear that a particle of $\frac{1}{8}$ inch will settle much more slowly than one of 2 inches diameter. Hence improved separation is to be obtained by careful screening and sizing beforehand. It is found that sizing is more necessary when cleaning by a pneumatic process than by water washing.

When it is necessary to wash very fine coal, say all below $\frac{1}{8}$ inch in diameter, it is often advisable to use a Draper washer, but this fine coal is difficult to dry, unless a centrifugal is installed. Wet washing is advantageously carried out by the usual Baum process, in which the coal and the dirt are jigged by a plunger, thereby allowing the dirt to sink to the bottom, where it is drawn off periodically. The coal flows over the weir with the water.

It is by no means certain that within limits the water contained in washed coal is actually disadvantageous to the manufacturer of furnace coke. Some authorities have stated that up to 8 per cent. of water is not so.

Dr. Koppers has investigated the heat required for carbonising different coals, and found that the heat necessary for dry coal is 9 per cent. greater than for coal containing 8 per cent. of water. He points out that a portion of the gases generated below 430° C. (the temperature about half-

way through the plastic layer) pass inwards. The sensible heat in these gases is sufficient to evaporate up to about 8 per cent. of water in the coal carbonised. Consequently, the first 8 per cent. of water evaporated requires no heat whatever in the oven flues, and the calculation of heat consumed must be based, up to 8 per cent. of water, solely on the weight of *dry* coal carbonised. The net result is that within this range, the drier the coal the more heat is required for carbonisation (E. M. Myers, *C.O.M.A. Year Book* 1934, 154). However, it can be said that coal with 10 per cent. of moisture has three times the volume of gases and vapours to be dealt with in the collecting main, and the condensation of this water vapour requires the abstraction of about four times the number of heat units to be abstracted in cooling the fixed gases, and the area of cooling surface must therefore be greater.

Dewatering and Drying of Coal.—Natural drainage cannot proceed beyond certain limits, dependent upon the amount of fine coal present. In America vacuum filters have been used for fine coal and slurries, and in extreme cases heat drying has been resorted to. In connection with froth flotation concentrates, it is stated that vacuum filters can dewater these down to 15 per cent., and if a lower moisture content is required, heat drying has been employed. In the U.S.A., at Pittsburg, there is a plant employing the following :—

(a) Coal down to $\frac{5}{8}$ or $\frac{1}{2}$ inch is dewatered by natural drainage.

(b) Centrifugal drying of coal between $\frac{5}{8}$ and $\frac{1}{48}$ inch.

(c) Heat drying of coal below $\frac{1}{48}$ inch.

It is said that the repair cost of centrifugals compares favourably with that of dewatering screens, and the extra power requirements may be considered justifiable in view of the extra water removed.

Coal Washing.—In coal washing there are many points to be considered :—the kinds of coal dealt with, their impurities, and the specific gravities of the materials. Some coals are more friable than others. In the same seam there may often be different kinds of coal.

The chief point to be aimed at is to wash the coal as thoroughly as possible, remove a maximum amount of impurity, and at the same time lose as little coal as possible. The analysis of representative samples of the material before and after washing will give a percentage elimination of mineral matter and sulphur. It should be remembered that the cleanest piece of coal that can be selected is not totally free from ash. The fact remains that some coals picked as clean as possible will still contain 10 per cent. or more ash. The coal passing away with the dirt should be rewashed carefully. Taking the specific gravity of the coal as 1·25, and that of shale as 2·2, then, by making a solution of calcium chloride or other substance with a specific gravity of 1·3, we have a liquid in which coal will float and shale, etc. will sink. Hence, if we take a weighed portion of the dirt from the washery, place it in the solution and stir well, the coal which floats can be skimmed off, placed in a filter-paper, washed free from calcium chloride, dried and weighed, from which the percentage of coal in the dirt can be found by calculation. In coal washing usually water is used, and we rely on the difference in the velocity with which various substances sink. The particles sink in water with a gradually increasing speed until they attain a constant maximum velocity given by the formula :

$$V = C\sqrt{d(s - 1)}.$$

It is seen from this that a large piece will sink in water at a quicker rate than a small piece, and pieces of coal, shale and pyrites will attain maximum velocities of 0·7, 1·41 and 2·56 ft. per second, a ratio of approximately 1 : 2 : 4. The falling velocity of a 2-inch piece of coal and

that of a $\frac{1}{2}$-inch piece of shale are approximately identical, and therefore the coal should always be carefully screened and sized before washing.

The principle of coal washing is to treat the coal with water so as to make the impurities sink to the bottom of the box, while the coal is kept above them. It is obvious that in the above extreme case of 2-inch coal and $\frac{1}{2}$-inch dirt this would not act. It is of great importance that the coal be correctly screened, and as a general rule the diameter of the largest particles should not exceed four times that of the smallest.

Dedusting and slurry washing are the chief improvements of modern years. Jig washers deal efficiently with nut sizes of coal, but less efficiently with the very small coal; for this, other forms of washers have been substituted. Dedusting or water clarification to clean slurry has been the greatest improvement. The human element has been reduced by the introduction of almost automatic control of the washing operation, by the use of shale-bed regulators with automatic dirt discharge for jig washers, and electric controls often with time-sequence switches.

It is now accepted that only a flotation unit can reduce the ash content of coal under 50 mesh I.M.M. ($\frac{1}{100}$ in.), and that it is unwise to pass this size to a wet washing plant. This size of coal if wetted retains 50 per cent. of the moisture after dewatering, does not mix uniformly with the other coal, and gives coke lacking a uniform structure. The only disadvantage of this dust is that it is generally high in ash, but it is usually rich in fusain and is thus useful to add to coking coal, helping to make stronger coke, and if added dry helps to lower the moisture content of the coal.

Dedusting should only remove the fraction of the coal which cannot be cleaned by ordinary methods, i.e. under $\frac{1}{100}$ in. This, however, cannot always be carried out in practice. Mott suggests that only coal below $\frac{1}{2}$ inch in size should be fed to a deduster, a preliminary screening at

$\frac{1}{2}$ inch in a modern vibrating screen being a useful method of shaking the dust from the larger sizes. The application of a current of air to the undersize will then have a reasonable opportunity of freeing it from dust. Mott further states that dedusting cannot attain the efficiency and sharp cuts which are desirable if the coal is high in surface moisture, and the use of hot air or flue-gas to aid the operation deserves encouragement. At least one firm has done this in practice, using air preheated at 550° C.

The practice of water clarification by the addition of flocculating media is now extensively practised and has developed rapidly in recent years. The reagents most commonly used are :—lime, starch, caustic starch, and proprietary reagents such as *unifloc*, a mixture of starch with calcium or zinc chloride or both. The most common practice is to make the water slightly alkaline with lime and then add starch with some caustic soda or some other reagent.

The method of dealing with slurry most widely adopted has been its settlement in a cone and its separate dewatering on a gauze screen, which tends to reject particles passing $\frac{1}{100}$ in., which are mainly clay, thus reducing the ash content ; while the use of concentrating tables and Rheolaveur slurry washers has effectively reduced the ash content of the material above $\frac{1}{50}$ in.

Dedusting should be the first practice to be applied to coal needing cleaning for coke making.

Dry Processes.—*The Birtley Type " Vee " Pneumatic Separator.*—This is a type of Birtley pneumatic separator for the dry cleaning of coal. It is an improvement on the " Wye " type of separator, from which it differs as regards the shape of its deck or surface (Fig. 1), but beneath this the essential parts are modified in detail only. The separator is reciprocated by means of eccentrics acting through toggles, these machines running at about 380 r.p.m. and having a

$\frac{3}{8}$ in. stroke. On the forward stroke the deck rises and on the backward stroke it falls, giving traverse of the material and helping to maintain the bed in a fluid condition. The air pressure below the bed of coal is maintained by means of a fan situated below the separator. Also below the separator deck is an air chest mounted upon the mechanism, and upon which the deck is in turn mounted, as shown in the sketch, with a hood over the whole machine.

Coal is first put into a bunker, whence it is fed into a
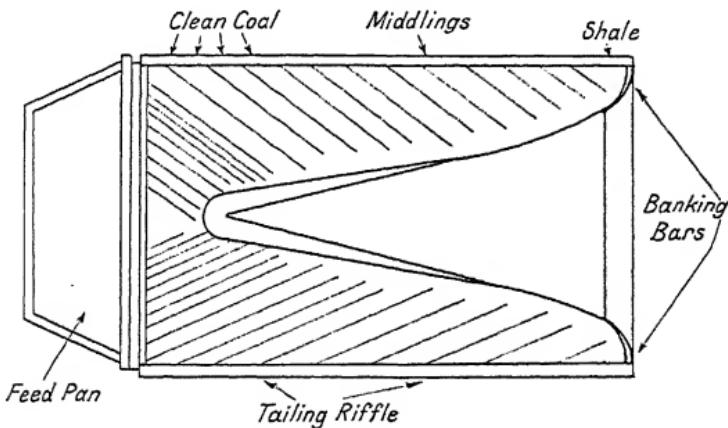
FIG. 1.—Birtley Dry Coal Cleaning Table.

boot of the raw coal elevator, by means of which it is lifted to the head of the plant. It is then screened ($1\frac{1}{2}$ in.–1 in. and $\frac{1}{2}$ in.–0), and lowered into storage hoppers above their respective separators.

The $\frac{1}{2}$ in. to 0 product is sized again over a hammer Bulldog Tandem Screen and two classes are hoppered and treated separately.

The coal is fed to the two larger sizes by mechanically operated shovel feeds, to the fines separator by means of an aspirator feeder, whereby the bulk of the fine dust is removed, and to the peas separator by a simple gravity

feeder with gate control. The separators are enclosed in mild steel hoods which are connected to the suction range leading to the dust filter.

Inspection doors are provided on each side of the separators so that the separation can be under continuous observation and any major fluctuations in the stone content of the raw coal followed up by the necessary adjustments of the cutting fingers.

The " middlings " consist of the overlap of clean coal stone, and these are collected from all separators on to one conveyor and returned to the raw coal feeder ; whilst the shale is similarly collected and loaded into wagons on a dirt road alongside the plant.

Each separator has its own fan and motor, power being transmitted to the separators by Renold chain drives and to the fans by belts.

The dust-laden air, after it has passed up through the coal on the separators, is evacuated through the mild steel trunking above the dust hoods by an exhauster fan and is blown into a bag filter. The air drawn through the aspirator contains a higher concentration of dust and also coarser particles than does the air stream passing off from the hoods ; it is therefore discharged into a cyclone which deposits the coarser dust and exhausts the finer particles over into the bag filter. The dust collected in the filter falls into two hoppers equipped with a common screw conveyor, which feeds the dust into the boot of an elevator. By this means the dust is lifted to the top of a dust bunker, from where it is conveyed pneumatically as required to the pulverisers and fed to boilers.

Referring to the plan of the " Vee " deck (Fig. 1), coal is delivered to the deck from the mild steel feed pan at the extreme left-hand side of the sketch and its distribution on the deck is controlled by two adjustable gates so that the necessary thickness of bed can be maintained across the width of the deck. The separating surface itself con-

sists of a heavy phosphor-bronze wire cloth which supports the bed of coal and is at the same time pervious to the air. The combined action of the air pressure and the jigging motion of the separator renders the bed of coal fluid, causing stratification to take place, the heavier particles being in the bottom strata and the lighter ones at the top.

Having stratified the raw coal into clean coal lying above a bed of refuse, it still remains to concentrate these products so that they can be collected in a state of purity. This is effected by moving the upper layer outwards and the lower layer inwards, whilst both are being propelled forward by the motion of the separator. In other words, the V in the centre of the illustration is acting as a plough and pushing off the upper layer of clean coal as the cross-section of the deck decreases. The refuse, on the other hand, is progressively deflected away from the clean coal spillage by means of the inclined riffles, which are tapered in height from the discharge end to the centre of the deck and are fixed at an angle of 15° to 20°, and is moved forward along the sides of the V until it approaches the curved end of the deck. Here, the last particles of the upper layer of coal having been squeezed off, the refuse crops out in a pure state and is banked up against the curved end of the banking bar before it is finally discharged.

In setting a machine to work, the inclination of the separating surface, the height of the tailing riffle, the speed of running and the pressure of the air are so adjusted as to maintain a fluid bed of coal over the entire deck. The operator soon learns to distinguish the correct position of the bed by throwing a handful of refuse on to its surface and seeing how quickly it sinks, or alternatively by putting his hand into the bed of coal.

R. Maxwell (*C.O.M.A. Year Book* 1935, 178) states that the substitution of a new type of deck consisting of thin steel plates having holes cup-punched in them, the size of

hole and deck opening being determined by the size of coal treated, has improved operating conditions.

The Static Dry Washer.—The static type of dry washer deals with unsized coal below 2 ins. or $1\frac{1}{2}$ ins. On this cleaning table the raw coal is stratified in a deep bed by a pulsating air current. The upper layer of clean coal is skimmed off the surface at three points in the 24-ft. length of the table, which is narrowed after each skimming to preserve the original depth of bed. In the fines dry-cleaning unit, the 15-ft. table is subdivided into six sections, at the end of each of which an orifice in the table allows the lower layer of dirt to be discharged, leaving the clean coal to flow eventually from the end of the sixth section. Cleaning is said to be effective down to $\frac{1}{16}$ in. in the 2 in. to 0 table, and in the fines tables clean coal of 6 per cent. ash has been produced from a feed of $\frac{1}{8}$ in. to 0, provided the surface moisture is kept below 3 per cent. Dedusting the raw coal is recommended and hot-air dedusting helps to bring the moisture content down.

Wet Processes.—*British Baum Coal Washing Plant.*— The Baum principle for the separation of coal from shale has been one of the best-known methods in this country for a number of years. The Simon-Carves " Auto-Constant " British Baum Washer Boxes have been built to deal with from 2 tons per hour to over 500 tons per hour through one unit.

The washing box (Fig. 2) consists of a strongly constructed steel tank with a cylindrical bottom. The tank is divided into compartments transversely and longitudinally. On one side of the longitudinal partitions are situated the piston air valves, which enable compressed air to enter the compartments at the back of the box. This compressed air acts on the surface of the water and forces it down so that the level of the water in the working compartments rises. In these there is a sieve plate extending the length
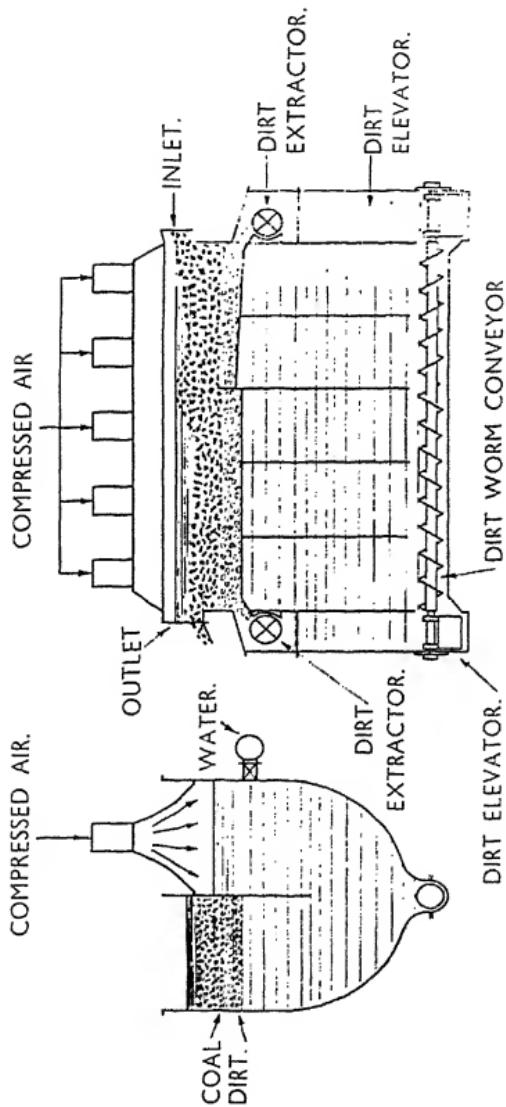
Fig. 2.—Baum Coal Washer.

of the box with perforations through which the water can rise, as mentioned above. The coal, which is brought by conveyor to one end of the box, is flushed into the box by water, and the rise in level of the water due to the action of the compressed air causes the coal to move upwards and forwards along the box while the heavier dirt remains on the sieve. The pure coal passes over a weir at the opposite end of the box and is then screened and dewatered as required. The shale thus rests on the sieve plate and forms a bed of a certain thickness. It will be readily understood that while the finer particles of shale can pass through the perforations in the sieve plate, the larger shale has to be extracted in other ways.

In the older plants vertical gates were employed for this purpose, and required hand adjustment to control the exit of the shale from each end. Close and continuous attention to the washing unit was thus necessary to obtain proper results.

The Simon-Carves system of automatic shale discharge has superseded the old method. It consists of rotating star valves, one of which is provided at each end of the working compartment of the washer box. The quantity of shale extracted is regulated by the speed of rotation of these valves. If the valves are stationary, no shale will be removed. The faster the valves rotate, the faster the shale will be extracted. When any particular coal is being cleaned, experience shows that the maximum efficiency occurs with a certain depth of shale. If the extraction of shale is regulated to keep a uniform thickness of bed, then separation takes place continuously at the highest efficiency.

The Simon-Carves system employs a float near each end of the working compartment of the washer box. This float is extremely sensitive and can take up a position according to the depth of the shale bed. The stem of the float is connected to a roller chain which passes over a wheel mounted on a shaft and fitted with two cams. This shaft is carried on ball bearings so that the friction is negligible,

and the cams work small poppet valves according to their position. These poppet valves allow compressed air at about 2 lbs. per square inch to pass into cylinders adjacent to the side of the washer box, one cylinder being provided for each star valve. Each cylinder contains a piston which connects to a lever operating on the shaft driving the star valve. If the piston moves the clutch into the " On " position, the star valve rotates and shale extraction takes place. If the clutch is in the " Off " position, no rotation of the valves takes place and no shale is extracted. It will be recognised that the star valve at the inlet side of the washer box has the more important rôle, as the shale content of the incoming coal may vary from time to time. In this case, therefore, a two-speed gear with a double clutch is fitted, so that if the float takes up a position corresponding to a shale bed of say 6 inches in depth, the lower speed is engaged and the star valve extracts at a certain rate.

Should the shale content of the raw coal be very high, then the shale bed increases slightly in thickness, by, say, 1 inch, and this causes the float to rise still farther and a higher speed is engaged with a consequent greater rate of extraction of shale. As soon as the excess shale is extracted the float falls to its normal position, and the extraction of shale takes place at, as near as possible, the rate at which it enters the box. Thus complete uniformity of conditions is obtained in the washer box throughout its period of operation, and the cleaning efficiency remains constant without human control.

The star valve at the outlet end of the box has little work to do unless there is a large proportion of intergrown ash or middlings present in the coal. In this case the valve then extracts the middlings and enables the ash content of the clean coal to be reduced to a minimum.

The use of compressed air for the production of the pulsations of the water in the box is, of course, common

to all Baum systems. The preservation of uniform conditions in the washer box by means of the " auto-constant " gear above described is exclusive to Simon-Carves, Ltd. It will be understood that the constant pulsation of water produces alternating stresses on the plates and connections which, if not properly taken care of, would result in a short life of the box and constant repairs. The whole platework is close riveted, of adequate thickness, and is securely braced and stiffened, to give the box a long life.

It will be realised that some means have to be employed to collect the smaller particles of shale which pass through the sieve as mentioned earlier. The usual method has been to provide a worm conveyor at the bottom of the box, which delivers the final shale to either or both of the elevators situated at each end of the box. In earlier types this worm conveyor was a source of some trouble owing to the wear that always occurred on the internal bearings which were necessary to support the worm conveyor throughout its length. These bearings could never be properly lubricated or inspected. In the modern washer boxes this trouble has been entirely overcome by the patented design of worm conveyor shown. This is the only type of shale collecting apparatus at the bottom of a " Baum " box with no internal bearings. As will be seen, the worm blades or scrolls are of cast steel and are bolted to lugs welded to a tube of sufficient diameter and thickness to avoid the necessity of any internal bearings. This patented arrangement has reduced the upkeep on the box to a minimum and is exclusive to Simon-Carves.

For compressed air, Simon-Carves' plants are provided with a high efficiency turbo blower running at about 3000 r.p.m. This machine requires no attention beyond very occasional lubrication. The rotor shaft is mounted on ball bearings, and there are no leathers or other wearing parts to renew. It produces an adequate supply of compressed air at a minimum consumption of horse-power.

Where a particularly difficult coal has to be dealt with the Hirst Patent Stirrer is employed. This stirrer consists of a carriage which holds a number of taper rods, the carriage being reciprocated transversely across a box by means of eccentrics. These rods disentangle any middlings or flats from the coal or the shale and assist in producing perfect elasticity in the bed. This device is the invention of Dr. Hirst of Birmingham University.

Modern practice in coal cleaning demands the utmost avoidance of degradation and breakage. It is often advisable, therefore, that the feed of the washing plant should come direct to the box without the interposition of any storage bunker or surge bin. Such a feed, therefore, will be fluctuating and the efficiency of separation can only be kept uniform if the pulsations of water in the washer box can be varied to suit the actual quantity of coal delivered to the box at any moment. To achieve this a third float is provided in the washer box ; this float rests not on the bed of shale, but on or slightly in the bed of coal, and it has been found that if the coal becomes too tightly packed, due to an overload, the float rises. On the other hand, if the input falls below normal the float falls. This motion of the float causes the pressure of the compressed air to vary according to the position of the float, and thus when the load is heavy, strong pulsations ensue until the load returns to normal, while if the load is below normal the pulsations are weakened to avoid loss of coal with the refuse.

Trough Washers.—*Rheolaveur Washer : Complete Modern Washery.*—A complete modern Rheolaveur washery consists of a large coal washing trough fitted with two sealed type Rheo-boxes, fine coal and slurry washing troughs, each fitted with open discharge type Rheo-boxes. In large washing troughs (Fig. 3), the coal to be treated is delivered to the head of the trough, where it is joined by a current
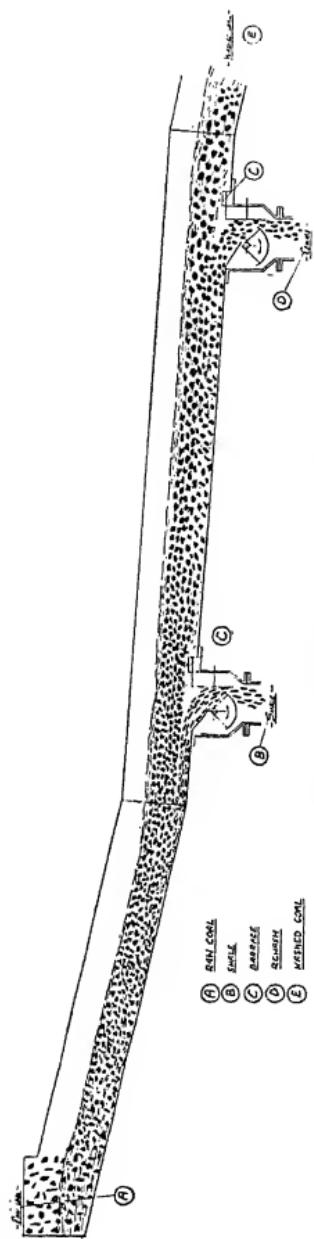
Fig. 3.—Rheolaveur Washing Trough.

A RAW COAL
B SHALE
C BARRAGE
D SLUICE
E WASHED COAL

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of water, and is passed down an incline to the first Rheo-box. Before reaching the first box the material is gradually and automatically separated, the shale falling to the bottom of the trough, and the coal being carried along by the horizontal current. A barrage is placed immediately after the first opening of the trough, the object being to create an additional lift to the coal over the Rheo-box opening, whilst the shale forms a bed and gradually falls through a slot in the trough to the Rheo-box. This first box is regulated so that only the heaviest material falls through the slot, and the coal passing over the barrage still contains a quantity of material having a high ash content. This in turn is separated in the second box, the material having the higher specific gravity, and incidentally the higher ash content, falling through the slot into the second Rheo-box.

The fine coal, together with the water, is extracted by a screen, and conveyed to the head of the fines washing installation, which consists of troughs fitted with open discharge type Rheo-boxes. The first boxes in the first and third troughs extract pure shale, whilst the other boxes extract a product containing the lighter shale and a small percentage of coal, which is delivered to the trough immediately beneath the boxes. The clean coal passing over the end of the two top troughs is passed to dewatering screens for disposal, while the material passing over the end of the remaining troughs is returned to the head of the first trough for retreatment.

Slurry Installation.—The slurry installation consists of superimposed troughs fitted with open discharge type Rheo-boxes, and the slurry goes through a similar treatment to the fine coal, with the exception that no upward currents are introduced. Where the material to be treated contains a large proportion of middlings, the main washing installation consists of two troughs, each fitted with two sealed type Rheo-boxes. The first box in the first trough evacuates a product which is conveyed to the head of the second

trough for retreatment, whilst the second box returns the material to the head of the first trough for rewashing. The first box in the second trough evacuates the shale, whilst the second box evacuates a product which is returned to the head of the first trough for rewashing.

The average consumption of power on a plant dealing with 75 tons per hour is 100 B.H.P. approximately. The Ashington Coal Company have a Rheolaveur plant designed to wash 285 tons per hour and it is said 416 tons per hour have been treated without affecting the washing efficiency, which was within the guaranteed figures (*Engineer*, **157**, 1934, 42).

Hoyois Washer.—The Hoyois form of trough washer has been introduced into this country from Belgium. The washing troughs are shorter than those of the Rheolaveur type. It is a modification of the trough washer, with upward currents separating layers of different gravity.

In the Hoyois small coal washer the coal and water are fed on to an inclined plate at the end of which there is a weir, through slots or holes in which a current of water cascades. The combined effect of this treatment induces stratification in a very short length. Subsequently the flow of water carries forward the upper layer of coal. The bulk of clean coal is thus recovered after passing through one trough 15 ft. long. At both ends of the three sections in this first trough are variable orifices or slots, beyond each being a weir of adjustable height. The heavier shales pass through these slots, up which currents of water pass. Clean coal passes over the end, and the rejects are passed to a second similar trough, where they are rewashed and the rejects again passed to a third trough. Thus the third trough separates all the shale from any remaining coal. The density of the material falling through the slots can be varied by the speed of the current. The troughs for different sizes of coal vary slightly in detail, but all act on the principle of washing and rewashing until finally clean coal is produced.

Density Washers.—Of the density types of coal washers, three distinct makes will be described :—the Barvoys, the Chance and the Tromp, using as media barytes, sand and iron oxide ore.

The Barvoys Process uses a medium of clay and barytes (barium sulphate), the clay being supplied by the coal itself and the barytes being so finely milled that a current of only 0·001 cm./sec. (or one-thousandth of that needed for sand) is necessary to maintain it in suspension. Coal is fed to a rectangular bath, and the floating portion is carried by a comb conveyor across the bath. The sinking portion is now dealt with by an upward current of medium in a compartment connected with the base of the flotation bath, so that middlings can be separated from heavy shale. This plant was first used at the Sophia Jacoba Colliery, near Aachen, in 1933 ; there are now ten plants on the Continent. The first in this country is at Wingate, Co. Durham. In this process, R. A. Mott says the cleaning is almost a static gravity separation, for the upward currents in the medium do not exceed in practice 0·03 cm. per sec. for coal of 3 ins. size. With an almost static bath and a well-designed comb conveyor, large coal can be treated without agitation and as easily as small coal.

Coal as small as 5 mm. or $\frac{3}{16}$ in. is cleaned in existing washers, and experimental washers have been used for successful cleaning to 1 mm. It is necessary to control the density of the medium by purging it from coal dust by a small froth flotation unit, and in practice the density of the medium is also controlled by small additions of barytes to make up for loss of medium with the products. The medium is easily sprayed from the clean coal, and the diluted medium is reconcentrated by settling in a thickener, the clear water being used as a fine spray for the finished products.

R. A. Mott states that the accuracy of separation due to the almost static conditions of operation enable an accurate

cut to be made at any specific gravity in a range which can probably be extended from 1·2 to 1·8, so that the percentage recovery of coal of a given ash content will be very high.

The Chance Process is one of the dense medium types, in which sand kept in suspension by an upward current of water is used. The sand is screened from the cleaned product, which requires to be screened at about $\frac{1}{16}$ in. before cleaning (R. A. Mott, *C.O.M.A. Year Book* 1938, 262). Coal up to 6 ins. or so can be fed unsized over $\frac{1}{16}$ in. to a simple cone, being carried round the cone by a light agitator and overflowing over a lip. The control is simple, no storage bunkers are required and the washing performance is good. Care is necessary to bleed slurry from the system, for it alters the effective specific gravity of the bath, and a continuous overflow of water is arranged to carry slurry to a sump. This washer has been used to produce coal of 2·5 per cent. ash content from Durham coal at Billingham, but the supporting current of water required for low specific gravities is excessive.

The Tromp Process.—The latest addition to the heavy-liquid types of washers is the Tromp process, which has been developed in Holland and is suitable for coal above $\frac{1}{4}$ inch in size.

Tromp uses a suspension of fine magnetite (sp. gr. 5·2) in water as the separating medium and the size of the magnetite particles (through 0·1 mm.) is so chosen that the resultant suspension is not completely stable. As a result of this the magnetite slowly settles to the bottom of the cone-shaped separating vessel. Thus there is a gradual increase in specific gravity of the liquid from top to bottom, say, 1·5 at the top to 2·0 at the bottom. The raw coal fed to the bath is quickly separated into pure coal which floats in the top liquid of lowest specific gravity, and heavy shale which settles to the bottom. Middlings, material of intermediate density, float in the middle of the

bath and their accumulation is prevented by the use of slow horizontal movement of the bath liquid, which moves the middlings to the side of the separator for removal by scraper conveyor.   There are three circulations of heavy liquid from three different levels in the bath, top, middle and bottom. The specific gravity of the top liquid corresponds to the cut between coal and middlings, say 1·5 specific gravity, and the bottom circulation to the specific gravity cut between middlings and shale, say 1·75 specific gravity. These liquids are returned to the feed end of the bath and are readmitted at the same level as they were withdrawn, thus producing the slow horizontal currents across the bath previously mentioned.   Provision is made for exchanging the liquids from any of these circuits as a means of control of the separating densities.

The products, clean coal, middlings and refuse, leaving the separator are washed free from magnetite, and these washings are fed into a small conical settling tank, where the heavy magnetite rapidly settles to the bottom and is returned to the system *via* a mixer, where the specific gravity is regulated.   Any fine coal and clay slimes also present in the washings settle on the top of the magnetite in this settling tank and are removed from the system.

The process gives excellent results, the ash contents of the products being practically those theoretically obtainable, whilst the losses of magnetite are small, about 1½ lbs. per ton of raw coal treated.   Due to the careful choice of grain-size of the magnetite, and the physical properties of magnetite being such that fine grinding does not produce much slimes, the equipment necessary for the recovery of the medium is simple in character.   The bath will deal with 70 to 80 tons per hour and there are at present two such plants working in Holland.

Froth Flotation Processes.—*The Minerals Separation Type.*—W. R. Chapman and R. A. Mott in their book *The*

Cleaning of Coal (London, 1928, 395–438) describe froth flotation. They say that the addition of a frothing agent such as cresol, aided by mechanical agitation, produces a bubble system in the liquid. A coal particle in contact with an air bubble is, in effect, equivalent to a solid particle at a liquid/air interface—the bubble will be stably attached to the solid—and if not too heavy its buoyancy will carry it to the top of the liquid, so that the coal particles will be concentrated in the froth. Shale, however, is easily dislodged from a bubble when agitated and is thus not borne to the surface. Therefore the separation of coal and shale is possible.

These authors say that, in the Minerals Separation plant, the coal crushed to a size less than $\frac{1}{10}$ in. is mixed with 4 to 5 parts of water, and is admitted to the agitation cell of the first box of a series. Here the reagents are added and paddles cause the necessary mixing. The agitated and aerated mixture then passes to the corresponding froth cell, where the first froth is collected. The residue passes from the bottom of the first froth cell to the agitation cell of the second box, and so on up to the last cell. At the discharge end a valve regulates the rate of flow through the plant.

The frothing agent added may be cresol, varying from $\frac{1}{2}$ to 5 lbs. per ton of coal, and the cleaned coal is recovered. By the addition at a later stage of a stabilising agent (neutral oil) in similar amounts, the middlings may be recovered in a stabilised froth. Chapman and Mott say that the drainage of the washed coal has proved the most serious stumbling block in the froth flotation process.

Minerals Separation plants have had a capacity up to 40 tons per hour.

The Elmore Vacuum Flotation Process.—The Elmore process is designed for cleaning fine coal below about $\frac{1}{8}$ in., and consists in mixing coal and water containing a trace of oil and applying a vacuum to the mixture. The coal attaches itself to air bubbles released from the water by the

vacuum, whereas the dirt remains unattached to the air bubbles. It is not a froth flotation process ; there is no froth in the Elmore process. The flotation is effected by individual coal particles attaching themselves to individual air bubbles which collapse and cease to exist as soon as they pass out of the vacuum to atmospheric pressure. It is the absence of a froth that enables the cleaned coal to be readily drained, and the cleaning to be done with a very low power consumption, as no agitation is required to produce a froth.

The plant consists of a double conical vacuum vessel and a mixing box. The fine coal, together with the required quantity of water and a trace of oil, is introduced into the mixer and the mixture produced falls into the feed tank and passes up the feed pipe into the washing vessel. The top of the washing vessel is connected to a vacuum pump, and as the feed passes up the feed pipe the air liberated from the water by the reduction in pressure forms a series of bubbles which attach themselves to the coal particles but not to the dirt particles. The attachment of the air to the coal particles is a physical phenomenon resulting from the fact that oil mixed into the pulp spreads itself on the coal surfaces but does not spread on the dirt. The air bubbles can only attach themselves to an oiled surface, and the coal particles, buoyed up by the air bubbles, rise to the top, whence they overflow into a launder and discharge pipe ; the dirt, being unattached to air bubbles, falls to the bottom of the conical washing vessel and is removed by a discharge pipe.

It is claimed that the ash content of the coal from this process is very close to that theoretically possible, and since the whole operation is free from mechanical appliances, except for water pumps and vacuum pumps, maintenance and running costs are low. The oil requirements vary from $\frac{1}{5}$ to 3 lbs. of oil per ton of coal and almost any kind of oil can be used, the usual oil cost being almost $\frac{1}{4}d.$ per ton. The cleaning capacity of one unit is about 5 tons per hour.

CHAPTER III

PRELIMINARY TREATMENT AND CARBONISATION OF COAL

Storage.—In an emergency, coals have been stored on the ground, but this is not very advisable, unless caused by necessity, as oxidation will take place and the coking quality of the coal will deteriorate. Further, if the coal is put in heaps on the ground, care should be taken not to pile it higher than about 18 feet, or it will have a liability to take fire by spontaneous combustion. Coking coals are usually stored in bunkers, which nowadays are generally built with reinforced concrete and are divided into different compartments. It is not advisable to keep coals in storage for longer than necessary, as in the course of time there is the liability of deterioration by oxidation. It is customary to have bunkers for the storage of two or three days' supply of coal for the ovens, but not usual to have bunker space for much longer. Should some special emergency occur, as the possibility of a coal strike, then the coke-oven manager would, of necessity, stock as much coal in the bunkers as he could, and put the balance in stock on the ground.

Blending.—At the coking plant the coal usually arrives in railway trucks, which on many works are bottom discharged by hand into a well. In some instances the railway trucks are mechanically tipped, perhaps by an electric cradle type wagon-tipper, incorporating a weigh machine. In modern works the coal is then transferred from the well by a belt conveyor to a blending bunker. In some older and

smaller works the blending has to be done in this first tipping well, perhaps by tipping the different coals together into the well, where they get partially mixed ; they are then elevated into the bunker, where the coals get a further mixing.   Where there is a blending bunker, the mixing can be more thoroughly and intimately carried out.

Blending of coals is always carried out to a certain degree by the coke-oven manager.   In an elementary way the manager mixes, say, two types of coal with which he is supplied and has to make the best possible coke.   Unless he carefully mixed or blended the different coals, the quality of his coke would vary.

Blending is carried out in the first place to improve the quality of the coking coals, and to make the best possible product with the raw material available.   Inferior or non-coking coals may be mixed with high coking coals, and a good coking coal may be the result.   Highly swelling coals may be diluted with others to make good coke.   Coke breeze or inerts may be added to improve the shatter index. The addition of fusain may make a more blocky type of coke.   The blending with, say, 5 per cent. of coke breeze, finely ground, may make a harder coke and reduce combustion, but it increases the ash content.   By hydrogenation it is possible to diminish the swelling power of highly swelling coal.

The need for the blending of coals prior to carbonisation in order to improve coke quality is being faced everywhere. In France, M. Berthelot has stated in a paper that in many cases the blending of suitable coking coals and some non-coking coals, prior to carbonisation, especially with cleaned coals, has led to improved coke, even better than that from the coking coal alone : while, in America, it is the general practice to mix two or more kinds of coal to improve coke quality, as many of the American coking coals if coked alone do not yield a satisfactory product.   In Japan the use of the Shimonura process, or the mixing of Japanese coal with

a semi-coke, previously carbonised at 400° to 600° C., has been the only possible method of producing good coke.

It is generally agreed that :—

(1) Blending considerably increases the range of coals available for coke production.

(2) The resultant coke from a suitable blend is often better than the coke from any of the individual coals.

(3) The heat penetration of a charge of blended coal may be improved, with consequent increase in output of a given plant and reduction in cost of production.

(4) Blending involves fine grinding of the coals.

Interesting work has been done with Saar coals and some weakly coking Silesian coals, when it was found that the strength of the coke produced was improved by a preliminary low-temperature carbonisation of a proportion of the coal and the intimate mixing of the low-temperature coke breeze with the remainder of the coal. In this case the proportion of the fusible constituents in the mixture of coking coal was much less than was the case with the raw coal, and yet the quality of the coke produced was better.

If the coals have to be washed, it has to be considered whether to blend before or after washing. If each of the coals contains about the same quantity of removable dirt and this quantity is likely to remain constant, an excellent method would be to blend dry and wash the mixture. It is not, however, generally possible to ensure that, even if the three coals have similar dirt contents when the plant is built, they will still have the same in a year or two. It may, therefore, be advisable to mix the washed coals.

If the coals arrive at the cokeworks by ropeway, bunkers should be provided for the dry coal, as only one type of coal can be washed at a time. Should they arrive by rail, shunting can usually be arranged, so that the washery can work on each coal so many hours per day. Assuming the

coals arrive by rail, each coal is first washed before being
elevated into a drainage bunker. Six drainage bunkers
should be provided and, if possible, have their discharge
outlets in a straight line, though this is not necessary where
suitable belt conveyors are arranged. These bunkers should
be of sufficiently large capacity for coals to be drawn from
bunker A while bunker B is filled with washed coal. Two
bunkers for each type of coal are sufficient if a coal dryer
is provided. If there is no dryer, it is preferable to have
three bunkers per type of coal going to the crushers.
At the outlet of each coal bunker is an adjustable sleeve
operating above a rotating turn-table. By adjusting the
height of this sleeve the amount of coal which issues
can be fixed. All the turn-tables are rotated at the same
speed. The coal will fall from all the turn-tables in opera-
tion on to the same belt conveyor, by which it is taken to
the dryer (if there is one) and then to the crusher.

R. A. Mott puts forward a theory of Professor Wheeler's
that the formation of coke involves wetting phenomena,
and that the strength of the coke depends not only on the
character and quantity of the fusible contents, but also on
the character of its non-fusible constituents. The more
easily these are wetted by the fusible materials in the coking
process, the stronger the coke.

G. E. Foxwell has stated that some coals contain a high
proportion of fusible compounds and still remain non-
coking ; apparently the attraction between the fusible
constituents and the surface of the coal is so weak that
binding does not occur. As the fusible constituents are of
a hydrocarbon nature, effective wetting is more likely to
occur if the non-fusible mass is low in oxygen, and here
appears to lie the explanation of the results obtained with
the Saar coals. Reduction of the oxygen content by the
preliminary low-temperature carbonisation of a proportion
of the coke breeze increases the capacity of the material to
be wetted by the binding particles, and thus the coke

strength is increased.   Similarly, the effects of fine grinding are accounted for by the suggestion that oxidation of coal is a surface action, and grinding increases the surface area and the capacity for wetting.   This may apply to blending also.

Hydrogenation.—Dr. Lander at the Fuel Research Station, Greenwich, made an important discovery of a method of increasing the coking power of coals by partial hydrogenation under pressure.   A coal, devoid of coking power, was powdered and given a mild treatment with hydrogen.   It came out of the bomb as a hard pitch-like material, which when carbonised made a hard swollen coke. In fact, it made a hard compact coke if mixed with two or three times its weight of the original non-coking coal. Further, it has been stated that a coking coal has been oxidised so as to lose its coking power completely, and has then been hydrogenated back to its original coking power. It is claimed that even anthracites can be converted into coking coals.   Similar results have been obtained using water gas instead of hydrogen.   As far as can be ascertained, this is the first recorded instance of the artificial production of the coking agglutinant in coal.

Crushing.—After blending, the coal is conveyed to the crushers, where it is usually crushed to a fineness such that at least 80 per cent. will pass through $\frac{1}{8}$-inch mesh.

Various types of crushers are used, generally in pairs, so that one machine can be used as a stand-by.

One modern type in use is the *Pennsylvania Hammer Mill Crusher*, which is of massive steel construction.   The rotors consist of heavy forged steel shafts, balanced steel discs and heavy hammer suspension bars on specially designed bearings.   The hammers, easily renewable, generally 152 in number, are of manganese or special heat-treated carbon steel.   These crushers have adjustable baskets comprising

breaker blocks and perforated plates, which can be adjusted according to the product required by wheel control during working. Such a crusher can deliver a product of 80 per cent. through $\frac{1}{8}$-inch mesh at 100 to 200 tons per hour, crushing coal 3 ins. and under, and using a 200 h.p. motor.

Another type is the *Jeffrey Diamond Swing Hammer Pulveriser*. These machines are worked with motors of 150 to 200 h.p. and up to 1200 r.p.m. They have cast-iron frames, with heavy-section renewable white iron liners, and removable hammers usually of some special carbon steel. They are capable of crushing up to approximately 125 tons per hour of 3-inch run of mine coal down to 80 per cent. through $\frac{1}{8}$-inch mesh.

The crusher rotor shafts are usually mounted in heavy type self-aligning roller bearings with mechanical forced feed lubricators. They are generally directly driven through flexible couplings. The crusher house is usually of reinforced concrete, built on a slab of heavy reinforced concrete.

Coke breeze may also be ground in special breeze crushers and fed into the coal crushers with the coal if it is found necessary to add coke breeze to improve the coke, but breeze passing through the crushers considerably shortens the life of the hammers in the crusher.

The crushed coal is then elevated up to the service bunker above the ovens, from where it is ready to drop into the coal-charging lorry for charging into the ovens. The service bunker is now generally built of reinforced concrete and will hold at least two days' normal oven requirements. This bunker is often made in a number of compartments, with at least one small one separate from the others, into which special coal may be put if necessary for test purposes.

The Carr Disintegrator type of crusher (Fig. 4) embodies four sleeves constructed of a large number of bars placed horizontally around the outer circumference. The outer sleeve revolves in one direction, the next in the opposite direction, the third in the direction of the outer sleeve, and

the fourth in the direction of the second sleeve. The material has to pass through the bars, and in so doing is crushed to the desired degree of fineness. This fineness is determined by the spacing of the bars. They are usually set so that over 80 per cent. of the coal is crushed to a fineness such that it will pass through an $\frac{1}{8}$-inch mesh.
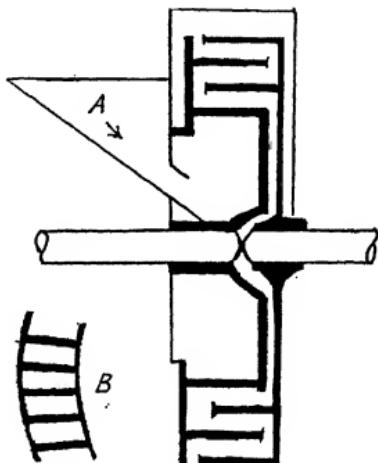

FIG. 4.—The Carr Coal Crusher.

A.   Coal feed.   B.   Section of portion of sleeve, with 1½-inch round bars spaced 2 to 3 inches apart.

Carbonisation of Coal

The transformation which occurs when coal is carbonised has been described by Porter as taking place in three steps, each involving its own set of reactions and having its own effects :—(1) the primary breaking down at low temperatures, liberating liquids and some gases and leaving a solid residue ; (2) secondary reactions between these liberated tars and gases at higher temperatures, involving both degradation and synthesis ; and (3) further progressive breaking down of the solid, producing chiefly gases and a hard carbonised residue. In all coal carbonisation processes,

as practically carried out, these steps are proceeding, in adjacent sections, simultaneously.

H. L. Riley (*C.O.M.A. Year Book* 1938, 309) describes the following picture of the carbonisation process : In the early stages of the pyrolysis of an organic substance, the process of aromatic condensation begins. The large flat molecules so produced arrange themselves in a layered structure similar to the graphite crystal lattice. As carbonisation proceeds, a temperature probably in the neighbourhood of 650° C. is reached, at which metallic electrons are liberated, this being accompanied by profound changes in the chemical nature of the coke. The graphite nuclei so formed still have hydrocarbon radicals bonded to the border carbon atoms of their hexagon planes. The high degeneracy of the hexagon planes suggests that these groups will be mobile. This would account for the crystallite growth which occurs as the temperature is raised to about 900° C. and the hydrocarbon groups are still further decomposed. Crystallite growth is continued only at much higher temperatures, depending on the nature of the carbon, by a process of thermal recrystallisation. It is probable that oxygen, nitrogen and sulphur atoms play important parts in the process of aromatic condensation, and their presence in the final coke has a considerable effect upon its properties.

The Destructive Distillation of Coal takes place when coal is heated in the absence of air, or in the presence of only a limited supply. During this process chemical reactions occur that are highly complex, and the new substances that appear in place of the coal are :—*gas, liquid* (water and tar) and a *solid* residue or *coke*. Coke, a porous, smokeless fuel, is the solid residue resulting from this destructive distillation of coal. The volatile matter is expelled by heat, leaving a residue, the character of which varies according to the nature of the coal treated, and according to the temperature to which the coal has been

heated.   The object of this carbonisation of coal is to obtain from it certain products, solid, liquid or gaseous, each having definite advantages over the raw coal in the industries in which they are to be employed.

In coke the ash content should not exceed 8 to 10 per cent., sulphur $1\frac{1}{4}$ per cent., phosphorus 0·02 per cent., while the moisture content in coke should not exceed 2 per cent. The ash content of coking coals is preferably 5 per cent., at most 7 per cent.   The volatile matter of South Wales coking coals may be as low as 18 per cent., while in Durham and Monmouthshire it is relatively low, from 25 per cent. to 30 per cent.   In Lancashire, one seam, the Lower Mountain Seam, has a volatile matter content of only about 20 per cent., and is the coal chiefly used for making the best beehive coke of that county.   Other Lancashire coking coals have over 30 per cent. V.M.   The coking coal of other districts is usually higher than Durham, from 30 to 35 per cent. V.M.

The carbon contents of many of the South Wales coals are similar to those of the low-volatile Continental coking coals, 88–91 per cent., and are usually much higher than those of other British coking coals.   Durham coals have the next highest carbon contents, 86–89 per cent. on the ash-free, sulphur-free basis ;   South Yorkshire coking coals have carbon contents from 84 to 87 per cent. on the same basis.   The oxygen content of all coking coals is less than 10 per cent.   The nitrogen and sulphur contents of Durham, South Wales and Monmouthshire coking coals are frequently less than those of other districts, and the yields of sulphate of ammonia are correspondingly lower, but the resulting cokes are of lower sulphur content.

The yields of by-products, for districts other than South Wales, Monmouthshire and Durham, are approximately 30 lbs. of sulphate of ammonia, 3 gallons of crude benzole and 7–9 gallons of tar, with 10,000 cubic feet of gas, per ton of coal.   In South Wales and Monmouthshire the

benzole yield is often only 2 gallons per ton, and the sulphate of ammonia only 20 lbs., while there is also a lower tar yield. The coke yield is higher than for other districts. In Durham the by-product yields are usually between the above extremes.

The coals low in volatile matter content yield a gas which is usually of lower calorific value than the gas evolved from coals richer in volatile matter.

The bulk of the coke produced is used in blast-furnaces, but the coke used at foundries and for other Home purposes has greatly increased. Oven coke is now used for water heating or steam raising in central heating systems in ever increasing quantities. It is used in steam road wagons, and in other boilers, for general heating and drying purposes in foundries (coil and mould drying), in bakeries (oven heating), in breweries (malt drying), in greenhouses and in closed stoves for house heating. This increased popularity of oven coke for uses outside the iron industries may be attributed to the increased manufacture of by-product coke in place of beehive coke, which was too hard and massive for most domestic uses, for which small coke of regularly graded size, or coke easily broken, is required.

The Thermal Decomposition of Coal.—A valuable paper on the chemical aspects of coal has been given by Lowry (*Amer. Inst. Chem. Eng.*, **30,** 1934, 280–298).

When a coal is charged into an oven whose walls are heated to some fixed temperature, the coal immediately in contact with the walls is heated extremely rapidly to the maximum temperature of the oven, while the coal at the centre of the charge is heated at a rate determined by the thermal conductivity of the mass and the temperature gradient, neither of which may be generally known. The intervening coal layers are heated at intermediate rates.

During the heating period, the coal passes through several poorly defined stages. The first effects of heat are com-

monly supposed to be the elimination of free water, and water and carbon dioxide by the decomposition of the more unstable groups of the coal itself. As the temperature is raised, the coal softens into what is frequently assumed to be, and is called, a plastic state. It has been suggested that this plastic condition results from the melting of constituents of the original coal, or of " primary " decomposition products, below their decomposition temperature and subsequent solution or dispersion of the solid residue. During the plastic stage, there is a more or less marked swelling of the coal accompanied by active evolution of the volatile, condensable components recovered in the tar, together with gases rich in hydrocarbons. As the temperature of the mass increases, the decomposition products become less and less rich in the condensable fraction and more and more rich in gases. The end of the carbonisation period in high temperature coking is marked by a shrinking of the residue, coke, and by an increasing ratio of hydrogen to methane in the gas evolved. The hydrogen in the gas is commonly attributed to the cracking of hydrocarbon gases. This may not be an entirely correct assumption.

Carbonisation Temperature and Products.—While 500° C. and 1100°. C. are roughly the temperatures of low and high temperature carbonisation, and their products may be divided into paraffinoid and benzenoid, yet there may be further differences between the higher temperature products at, say, 1350° C. and those obtained at the lower temperature of 1100° C. C. B. Marson and H. V. A. Briscoe (*C.O.M.A. Year Book* 1933, 190) have given some particulars. Parker and others have shown that increase in the carbonisation temperature increases the amount of gas made and the gaseous therms per ton, but decreases the tar and ammonia yields. High temperature tars are characterised by greater amounts of free carbon and lesser amounts of tar acids. High temperature also favours the production

of naphthalene. Seelkopf, as a result of laboratory investigations in which Lower Silesian, Ruhr and Upper Silesian coals were examined at coking temperatures varying between 900° and 1200° C., found that high coking temperatures yielded higher proportions of pitch and lower proportions of light oil constituents.

S. Tweedy states that short carbonising times (and hence presumably high flue temperatures) increase the benzene content of benzole, with a corresponding decrease of other constituents ; he points out, however, that the greater amount of benzene recovered is not necessarily due to the higher flue temperatures, because the composition of coke-oven benzole may be influenced by any of the following factors :—type and size of oven, carbonisation temperature in the oven, uniformity of heating, space occupied by the charge, class of coal, including its moisture content, and general operation of the oven. S. Tweedy believes that in modern batteries with larger and narrower ovens, the cracking effect is reduced to a minimum, and although the shorter carbonising times in some ovens produce a benzole of high benzene content, the yield shows no decrease and the combined effect on the total yield of by-products is not a serious matter ; in any case, the benzole produced in modern ovens is often of better quality.

S. Tweedy's conclusions are directly opposed to those of Kuhn, who describes a recently built coke-oven battery, with ovens $17\frac{1}{2}$ ins. wide. On certain occasions, when dry, good quality coal was coked with a wall temperature of 1450° C. or over, the coking time did not exceed 13 hours. Under these conditions the saving in capital was considerable ; on a battery of between 48 and 69 chambers, where the coking time was reduced from 26 to 18 hours, the yearly saving worked out at £10,500. The yield of by-products was reduced, however, and a careful calculation showed that the entire saving effected by this reduction of coking time was more than offset by the loss of by-products. Rapid

COMPARISON OF PRODUCTS OF HIGH AND LOW TEMPERATURE
CARBONISATION OF COAL

	High Temperature Carbonisation (1100°–1300° C.).	Low Temperature Carbonisation (500° C.).
Coke.		
Percentage of volatile matter	about 1	about 10
Tar.		
Gallons per ton of coal .	10–12	up to 22
Percentage weight of coal	5	10–12
Specific gravity	1·2	1·08
Chemical nature . . .	benzenoid	paraffinoid
Gas.		
Cubic feet per ton of coal .	10,000–13,000	4,000–5,000
Percentage composition :—		
Hydrogen	48·0	25·0
Methane	27·0	40·0
Benzene	1·0	20·0
Other hydrocarbons . .	2·5	6·0
Carbon monoxide . .	9·0	4·5
Nitrogen	9·0	2·0
Carbon dioxide . . .	3·5	2·5

throughput, therefore, may not necessarily make for economy.

The rate at which heat is transmitted to the charge may be influenced by :—

(1) The width of the oven.

(2) The flue temperature.

(3) The coking quality of the charge.

(4) Its moisture content.

(5) The nature of the refractories forming the walls.

A reduction of oven width usually, but not necessarily, corresponds with an increase in the rate of heating. With regard to the relation of coking time and oven width, the old rule that the coking time in hours equals the width of

the oven in inches is a very crude approximation, indeed is often inaccurate.

Gas.—The most modern by-product from coal, now considered one of the most valuable, is the gas after it leaves the benzole scrubbers of a modern coking plant. The increase in the building of regenerative ovens, in which some 60 per cent. of the gas produced was available for sale, and, further, the adoption of the compound oven, where the ovens themselves can be heated either by a portion of the gases produced at the plant itself or by blast-furnace or producer gas as required, have made available for sale the whole of the gas generated from the coal carbonised.

The methods of disposing of surplus coke-oven gas may be set forth as follows :—

(1) Sale to public gas supply undertakings.
(2) Disposal for industrial purposes, steelworks, etc.
(3) Power generation, gas engines.
(4) Steam generation.   .
(5) As a source of ethylene, methane and hydrogen.

In notes relating to the supply of coke-oven gas for public purposes, submitted on behalf of the Coke Oven Managers Association to the area Gas Supply Committee of the Board of Trade in 1929, D. V. Hollingworth stated that there would be an estimated amount of 13,787 million cubic feet of coke-oven gas supplied from coking plants in this country for public purposes in 1930. Since then, more coke-oven plants have begun supplying gas to public gas undertakings, and the amount should now be a good deal higher. Those notes point out the suitability of coke-oven gas for public supply, its reliability and flexibility of supply.

Generally, the coke-ovens supply the gas unpurified to the gas undertakings, but in some instances the gas is purified at the ovens. The gas industry requires gas in

variable quantities. In the summer only a small quantity is needed ; then the coke-oven battery supplying the gas would probably have all its ovens heated by oven gas and the ovens would work with the least possible throughput. In the winter, when a large load is required, the ovens would be worked to their utmost capacity, and would be heated by producer or blast-furnace gas, thus liberating an additional 40 per cent. of coke-oven gas for sale. Usually the gas is required by the gas undertaking at a stated calorific value, between 475 and 540 B.T.U., and with a naphthalene content below a certain figure.

Benzole.—Owing to the enormous increase in motor traffic, and the improvement of the internal combustion engine, the supply of motor spirit to a specification laid down by the National Benzole Association has become not only of national importance, but also a source of profit to the coking industry. There is a greater demand than a supply of this product. Benzole is extracted from the gas by absorbing it with oil, from which it is distilled, rectified and purified ready for sale. The method of its extraction and purification is described later.

Ammonia.—This is recovered in the coking industry in quantities up to 35 lbs. of sulphate of ammonia per ton of coal carbonised. Whether extracted as sulphate or as 25 per cent. ammonia liquor, ammonia has been an important factor in the success of the coking industry of the past decade. Now, owing to lower sale price, perhaps brought about by the competition of synthetic sulphate of ammonia, there is very little profit in making this by-product. Ammonia in the form of chloride, as liquor or as purified salt, is also a recoverable product on plants employing the " direct " method of ammonia recovery. The actual amount produced varies with the chloride content of the coal.

Tar.—Coke-oven tar is a mixture of very many different compounds condensed from the gas. It varies according to the coal used and the temperature at which the coal is carbonised. It is a black, rather thick and viscous liquid, 12 to 22 per cent. heavier than water (its specific gravity varies between 1·1 and 1·2), and usually semi-solid when cooled to 0° C. It is a valuable product to the tar distiller and for sale as road tar when refined. Dehydrated and refined tar is being required more and more for the modern motor road, and as such is a source of value to cokeworks. When distilled it yields anthracene, carbolic acid, creosote and light oils, and gives a solid residue, pitch.

It is said that high tar yields are obtained by carbonisation with rapid rates of heating, while low rates of heating tend to give increased coke yields at the expense of reduced tar yields. Higher temperatures of carbonisation tend to thicken the tar and give it a high pitch content, with a low phenolic compound content, more naphthalene and anthracene, and more free carbon, with more oils of a high specific gravity. The efforts of the British Road Tar Association are increasing the use of tar for road surfaces. The superior adhesion qualities of tar are specially suitable as a road binder, and tar is being more and more used by road engineers.

Naphthalene.—Naphthalene, $C_{10}H_8$, is a product of high temperature carbonisation, high temperature tar containing from 4 to 10 per cent. of naphthalene. It melts at 79° C. when pure, but impurities greatly reduce this melting-point ; its boiling-point is 218° C. Above 1020° C., benzole gives place to naphthalene ; if the products of carbonisation are not cleared away quickly from the hot walls at the top of the oven chamber, naphthalene increases, and this decreases the yield of benzole due to the gases cracking.

Naphthalene is sold either (1) in a purified form, or (2) as crude naphthalene. It may be sold as pressed naphtha-

lene, having been converted into cake form in a press at a pressure of approximately $2\frac{1}{2}$ tons per square inch. These cakes are broken into small pieces and bagged for sale. Naphthalene in crude form is used in the manufacture of fire lighters (in some instances as a mixture containing 50 per cent. of sawdust). It is also employed as a germicide, in the manufacture of lampblack and as a solvent for indigo, and is the source of synthetic indigo. Naphthalene is used in the dye-industry and in the manufacture of explosives, and it has been utilised as a fuel for internal combustion engines.

On a coking plant the bulk of the naphthalene is removed by cooling, and the balance can be removed by washing with oil. Gas sold to gas companies for public use usually has to have the naphthalene content below 2 grains per 100 cubic feet. Using a naphthalene oil washer no difficulty should be experienced in reducing below this content. Some form of direct cooling has been found to be the best for removing naphthalene, either water or oil having been in use for this purpose. The solubility of benzole in water is about 0·1 per cent.; Dr. Foxwell (*C.O.M.A. Year Book* 1938, 145) considers that even 0·1 gallon per ton is worth saving and suggests that it should be looked into whether direct cooling with water is the best practice, while naphthalene itself is an excellent solvent for benzole.

CHAPTER IV

COKE-OVENS

Refractory Materials for Coke-oven Construction

Silica Material.—During the past fifteen years a great change has come over the minds of those building coke-oven batteries in this country. Instead of using semi-silica material for coke-oven construction, silica material is now used almost exclusively for the walls, flues and regenerators of modern ovens. It is manufactured from a certain type of hard quartzite or ganister containing approximately 98 per cent. silica. This material is finely crushed and bonded together with a small amount, usually 2 per cent., of CaO in the form of hydrated lime as fluxing agent.

When a high-quality properly burnt silica shape is heated under load no softening or distortion occurs until a temperature of at least 1670° C. is reached, absolute rigidity being maintained right up to the point of collapse, which is nearly 300° C. higher than that at which it is safe to use other materials in coke-oven walls (A. H. Middleton, *Coke Oven Managers Year Book* 1926, 401).

Another advantage is the greatly increased throughput that can be obtained with silica, due to its better conductivity and the higher temperature that its use makes possible without risk of damage due to overheating.

Silica is subject to two expansions :—(1) permanent (2) thermal or reversible.

Permanent expansion depends solely upon the time and temperature attained while burning the material at the

brickworks. If properly burnt, this expansion has taken place already at the brickworks. An unburnt brick consists of silica as quartz (specific gravity 2·65) ; on heating to a higher temperature for a certain period, this changes to the less dense forms, cristobalite (specific gravity 2·33) and tridymite (specific gravity 2·28). If the silica material is properly burnt, the whole of the quartz is converted and no further expansion from this cause may be expected. This expansion is permanent and not reversible.

In addition to permanent expansion, there is the ordinary thermal expansion which is reversible and common to practically all materials. When silica is heated from ordinary temperatures up to 1600° C., this expansion amounts to approximately 1·3 per cent. (in fireclay it is 0·75 per cent.). The greater part takes place under 300° C. and some at about 575° C. In heating up a battery of coke-ovens, the expansion of the silica material is usually allowed for by having expansion joints filled with sawdust and topped off with pitch.

In silica ovens temperatures up to 1400° C. can be allowed ; this means that up to 1500° C. is occasionally attained in spots. The use of silica has made the modern oven possible, enabling high ovens of 15 to 18 ft. to be used, at higher carbonising temperatures, and carrying a load of heavy charging machinery. With semi-silica the lower courses of the then 7 ft. high ovens occasionally used to become deformed owing to squatting. In almost all recently built coke-ovens, silica has been used for the superstructure at least, and the experience of the past fifteen years has shown this material to be satisfactory. The price of silica material is higher than that of semi-silica, but it gives greater durability, much longer life with less repair costs, not to mention a greater margin of safety. It has greater flexibility of temperature, the possibility of much greater output and, finally, greater resistance to the corrosion of salty coals.

Semi-silica or Half-ganister is the material with which the walls of all the older ovens were built ; it is midway between fireclay and silica in properties and has a silica content up to 84 per cent. This material may occur naturally as a ganister or is made by adding ganister to fireclay. The refractoriness is usually lower than that of either fireclay or silica, but it does not commence to distort under heat and load at such a low temperature as fireclay. Complete collapse of the best of these mixtures occurs at 1400° C., which leaves little margin of safety under modern temperatures, owing to the impossibility of avoiding peak temperatures above the capabilities of this material. It may be used for the regenerators of ovens in some instances, although silica is more generally used.

Fireclay is the cheapest of the refractory materials available ; it is made from natural clays with a silica content of 50 to 65 per cent. and an alumina content of 27 to 40 per cent. Of these three, fireclay probably withstands rapid variations in heat at comparatively low temperatures the best, but it is unsuited for the hotter parts of the oven owing to its inability to withstand *high temperatures under load*.

Fireclay material can have its melting-point lowered considerably under pressure, which explains the distortion that often occurs in the lower courses of coke-oven walls built of clay-bonded semi-silica material.

A good fireclay has a refractoriness or fusing-point of 1700° C., which is near that of silica, but although it may not itself melt under 1700° C. it does, under heat and structural load, begin to contract, soften and lose shape many hundreds of degrees lower. It is therefore only used in coke-oven construction where it does not get much heat. It may be used in the substructure, possibly in the regenerators, and as a paving on the top of the oven structure. A hard-burnt fireclay is usually used for the soles of the oven.

Systems of Heating Ovens

The by-product oven into which the coal is charged is a long, narrow, oblong chamber from 30 to 40 ft. long, 14 to 24 ins. wide and from 7 to 18 ft. high, with heating flues on both sides.

The main problems of carbonising are :—

(1) to keep the heat in the flues at a steady temperature, at from, say, 1000° to 1300° C., as may be required ;
(2) to decide beforehand what should be the correct proportion of the oven width ; and finally,
(3) to ensure uniform heating of the charge.

Carbonising engineers and oven designers have been studying these points for the past quarter of a century, and are still seeking further improvements.

The first types of Semet-Solvay and also Simon-Carves ovens were heated by horizontal flues running along each side of the oven charge. Later, vertical flues were adopted in the Otto and other types of ovens. These older ovens were generally of the waste heat type, *i.e.* all the gas generated, after passing through the condensing and by-product plant, was returned to the ovens and burnt in the flues to heat the charge. The products of combustion, which may previously have been allowed to go to waste up a chimney to the atmosphere, passed along a flue to the boilers, where this so-called waste heat was used to raise steam for use on the coking plant or at an adjacent colliery, or to generate electricity.

Later, it was found that gas was a salable product either for use at a steelworks, or for sale to public gas supply undertakings. Thus the regenerative type of oven was evolved. Coke-oven flues are heated with gas burnt with the correct amount of air. In a regenerative oven the greater proportion of the heat in the spent flue-gases is transferred to the air supply. Thus a less quantity of gas is required to maintain the oven temperatures and a greater

proportion of gas is rendered disposable for purposes not directly connected with the coking plant itself, such as gas lighting, gas engines, etc. On the other hand, the transference of heat from the spent gases leaves them just hot enough to create the necessary chimney draught, and no heat is left for boilers or other purposes.

Of the heat units represented by the gas evolved, the surplus is available either in the form of live gas or as waste heat. Choice between the two should be influenced largely by geographical conditions. If the plant is in close proximity to a town requiring gas, an oven giving a great proportion of spare gas would probably be the best. If not near populated areas, but near a colliery requiring steam, a waste-heat oven for steam-raising purposes would be suggested.

Reversing Principle of Regeneration.—Suppose the products of combustion rise in the left-hand flue section and descend the right-hand section (Fig. 5). This cannot go on indefinitely, otherwise the cold air would gradually cool the chequer work B to atmospheric temperature and the oven would be a waste-heat oven, and require more gas to heat the colder air for combustion. Consequently, about every half-hour the damper E is reversed, while all gas-cocks at G are opened and those at F are shut. The new direction of air and gas is now anti-clockwise, and the air for combustion is now preheated.

The modern regenerative oven was evolved, in which approximately 60 per cent. of the gas generated from the ovens was available for other purposes than heating the ovens themselves. The use of producer gas for heating ovens came into use in the compound oven. This had been in use at gasworks for many years, chiefly for the reason that here gas was the main product, and any notion of heating a retort with coal-gas was not to be thought of for a moment. On the other hand, the gases from coke-ovens

were originally of so little value that the use of a portion
of the then valueless gases was deemed sound economics.
The compound oven using coal-gas, or either producer gas
or blast-furnace gases, as occasion demanded, became an
economic proposition when gas undertakings began to
purchase coke-oven gas in large quantities. If the coke-
oven gas can be sold or used, the whole of the gas generated
from the coal is set free for that purpose. When there is
an adequate market for coke-oven gas, the possibility of
increasing the normal yield by some 40 per cent. is extremely
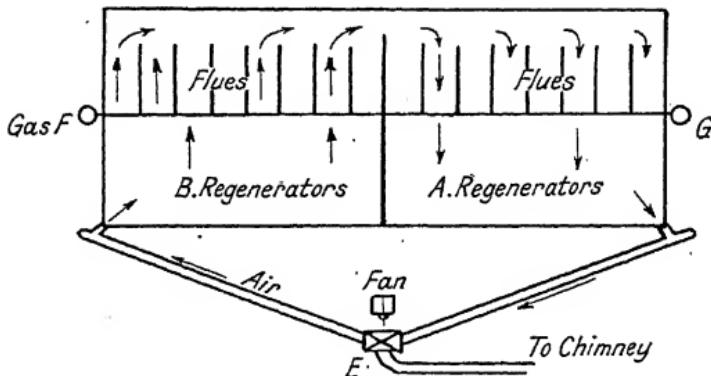
FIG. 5.—Reversing Principle of Regeneration.

important. Messrs. Koppers stressed these points and have
emphasised these advantages. Further, if some of the gas
can be sold, say in the winter months and not in the summer,
it is a simple matter to heat part or the whole battery with
producer gas as required. By using an outside mechanical
producer, the breeze can be used for heating the ovens and
thus a product not readily salable can be utilised. Finally,
the oven could thus be kept hot during a strike period
without the necessity of charging any but the few ovens
necessary to provide coke for the producer.

There are a number of excellent types of modern coke-
ovens, but this section will be limited to a short description

of the Becker, the Otto Underjet and the Koppers Compound Regenerative ovens. Of other types on the market, it may be mentioned that the Still oven is a high oven which is heated by having the gas entering at several ports at intervals up the vertical flue at varying heights, thus ensuring a very long flame. The Collin type, however, has continuous heating of all the vertical flues with alternate " up " and " down " combustion, resulting in the heating of the whole oven-wall surface by the actual flame. The air is supplied to the top flue by a special flue which becomes a waste-heat flue when the gas is burning from the bottom flue.

Combination Ovens Heated by Coke-oven Gas.—

Koppers Compound Regenerative Oven.—The Koppers Circulation Oven is for ovens and combination ovens heated by low-grade gas ; the flow of the gases is illustrated in Fig. 6. The design of the oven chamber, the arrangement of the charging holes, gas off-takes and so forth, call for no special comment, and attention may, therefore, be concentrated upon the disposition of the flues and regenerators and upon the flow of air and gas through the heating system.

Air enters at the inlets A (known as " knee " pieces) and is distributed beneath the regenerators by the ducts B. After rising through the regenerators situated on the right-hand side of the battery, a portion of the air flows to the flues on the right-hand side through six ports C situated at the top of the regenerator chamber. The remainder of the air flows through a short diagonal duct D into a horizontal duct E, from which it passes through ports F, and by channels G into the flues H.

When the coke-oven gas is used for heating, it is fed from a main I into the horizontal gas ducts J, and meets the air at a point slightly above the sole of the oven. If it is proposed to use producer or blast-furnace gas for heating the ovens, the gas is fed into the alternate pairs of regener-
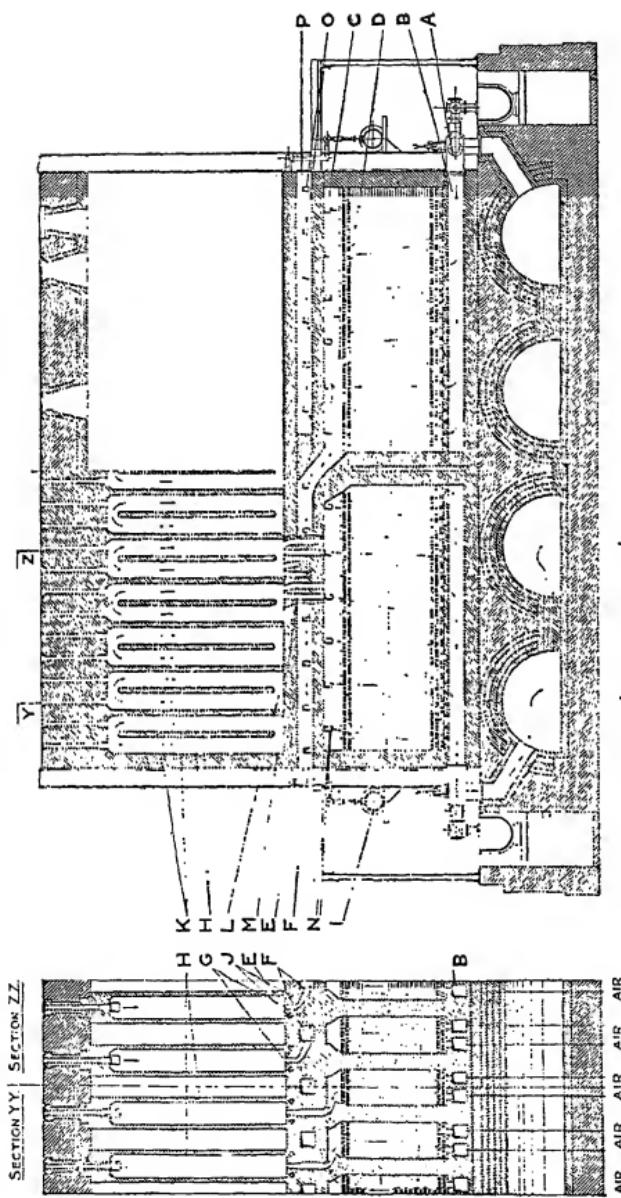
FIG. 6.—Koppers Circulation Oven.

(a) Combination Oven for Heating by Coke-oven Gas.

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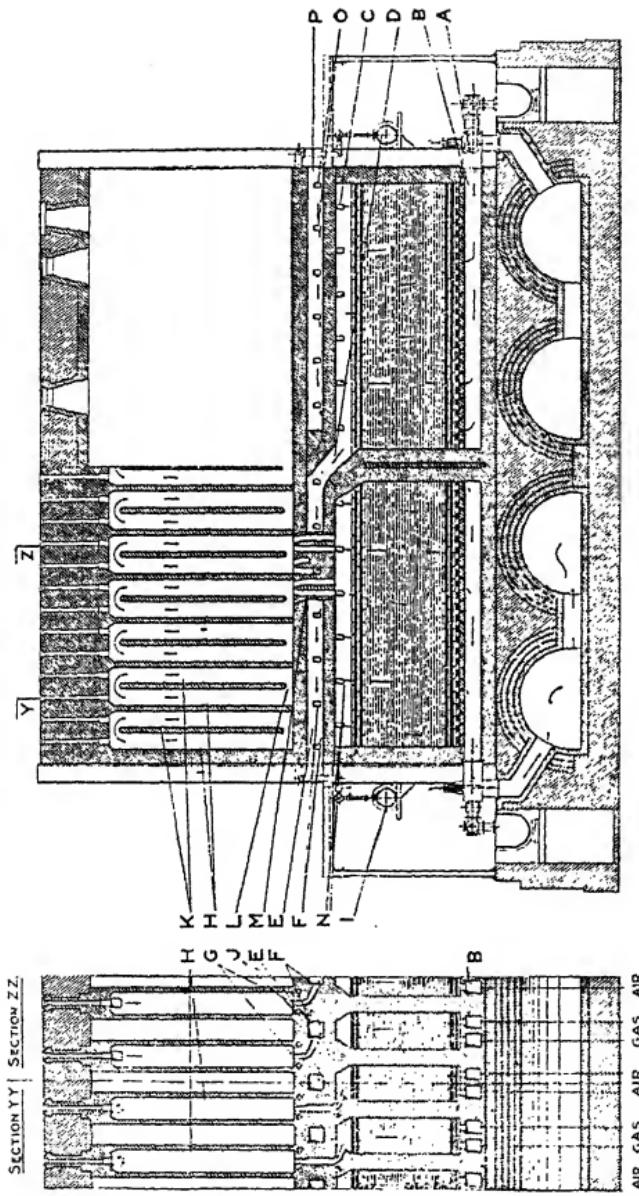
Fig. 6.—Koppers Circulation Oven.

(b) Combination Oven for Heating by Producer or Blast-furnace Gas.

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ators situated collaterally with the air regenerators just described. The travel of the producer gas is exactly similar to the travel of the air.

The flues are arranged in pairs (or "twins") H,K, separated by a mid-feather. At the bottom of this mid-feather is the circulation port, consisting of an opening L, forming the duct through which a portion of the waste gases is drawn from the downtake flue K into the heating flue H, for the purpose of lengthening the flame. The gases burn upwards in the alternate flues H, and the products of combustion pass over the mid-feather as shown by the arrows and down the second constituent of the twin flues K, leaving the flues by channels M.

The gases from the flues situated on the left-hand side of the battery pass by ports N directly into the left-hand regenerators. The products of combustion from the flues on the right-hand side of the battery flow through ports O into the horizontal duct P, issuing thence downwards through a diagonal duct D, shown dotted and approximately at right angles to the similar duct D previously described, to join the rest of the burnt gases at the top of the left-hand regenerator. The hot gases then pass down this regenerator, where their sensible heat is recovered in the usual manner, and enter the waste-gas flue, from which they are discharged into the chimney. The direction of the gas and air is reversed in the usual manner half-hourly, so that the air then enters the base of the left-hand regenerator, and the gas burns upwards in flues K, while the burnt gases flow down the flues H, into the right-hand regenerator.

The Woodall-Duckham Becker Combination Regenerative Oven.—The basic feature of the Becker coke-oven is the cross-over flue. This arrangement makes it possible to employ horizontal flues of small dimensions in spite of the big volume of gases to be handled, thus ensuring great strength of battery structure, even heat distribution at

every point of the oven-wall, and high thermal efficiency (Fig. 7).

The heating system of the W.-D. Becker oven consists of a series of vertical flues on each side of each oven, with two regenerators built under each series of vertical flues. When employing blast-furnace or producer gas as the heating medium, air travels up one regenerator and gas up the second regenerator. The air and gas meet in the vertical combustion flues, up which the gases travel in parallel. The gases are collected from each four or five vertical flues and are carried over the top of the ovens by means of cross-over flues. There are six cross-over flues to each alternate oven. The intermediate ovens have no cross-over flues. From the cross-over flues the waste gases are distributed into vertical flues exactly opposite the flues up which they have travelled on the other side of the oven. They travel down the vertical flues and are equally distributed in the two regenerators below. This arrangement of heating ensures that the travel of gases is short and the speed low, so that excellent regulation can be obtained with a low pressure differential. The heating gases in all the vertical flues on one side of an oven always travel in parallel flow, and there is no possibility of short-circuiting and consequent loss of efficiency.

On the other hand, when employing coke-oven gas as the heating medium, both regenerators are used for preheating the air for combustion. The coke-oven gas is delivered through a horizontal gas conduit carried in the brickwork between the top of the regenerators and the bottom of the vertical flues. From these conduits the gas passes by means of a calibrated nozzle and vertical duct into the bottom of each vertical flue, where it mixes with the preheated air. The air and gas come together with a streamline effect and the combustion zone is evenly distributed throughout the height of the vertical flues. The products of combustion are then collected from the top of each group of four vertical
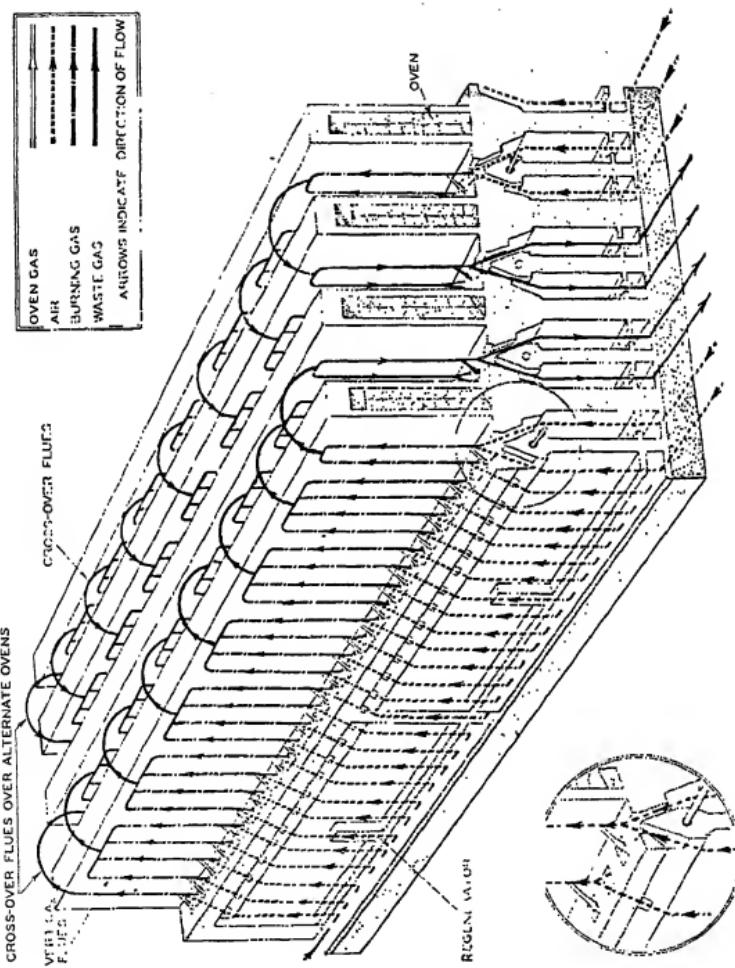
FIG. 7.—Four Coke-ovens of the Becker Type.

flues and are carried over the top of the oven by means of cross-over flues. There may be six or eight cross-over flues to each alternate oven, none being required for the intermediate ovens, as may be seen from the illustration.

From these flues the waste gases are distributed into an equivalent number of vertical flues on the other side of the oven, exactly opposite the flues up which they have travelled. They then travel down these flues and into the two regenerators immediately below, through which they pass to the waste-gas flues, giving up heat to the chequer filling in transit. Upon reversal, which is automatically controlled, the air travels up these regenerators, through which waste gas has previously passed, and meets the coke-oven gas, and combustion takes place upwards ; the products of combustion travel through the cross-over flues, and down the flues up which the burning gas has previously travelled, finally passing out through the corresponding regenerators.

Although, as in other types of ovens, more gas is applied to the wider end than the narrower, in order to carbonise the varying width of coal charge in the shortest time, maximum efficiency of regeneration is obtained in the Becker system, because the quantity of air at any point in the length of the oven takes up from the regenerator brick-work the heat which has been given to it by the waste gases from an equivalent quantity of air and gas mixture. By employing the principle of the cross-over flue in the construction of the battery, great strength is obtained, inasmuch as all horizontal flues or conduits are reduced to a minimum in number and in cross-sectional area. The reversing valves are designed so that, with the provision of blast-furnace gas mains, any or all of the ovens can be heated by means of blast-furnace gas.

Simon-Carves Underjet Regenerative Compound Type Oven.—This type of oven is of the Otto compound twin-flue design, that is, any number or all of the ovens may be

heated by either blast-furnace gas or coal-gas, the change-over from one to the other method of heating being effected by the simple manipulation of valves on the fuel-gas supply mains (Fig. 8). Thus, if needed, the whole of the gas generated during carbonisation is available for sale for industrial use when the ovens are heated with blast-furnace gas.

The essential features of the Underjet system are :—

(a) The external distribution of rich fuel-gas, which enables the quantity of gas burnt in each heating flue to be accurately controlled.

(b) The uniformity of pressure along the whole of the heating walls due to the absence of unbalanced bus flues, which ensures adequate distribution of the air and/or lean fuel-gas to the various heating flues without the complication of internal regulating devices. Consequently the heating of the oven is extremely uniform and the charge is evenly coked. The only regulating points are adjustments of the fuel-gas pressure and the chimney draught.

Under each oven is a regenerator chamber divided longitudinally into halves on the centre line of the battery. Each half-regenerator is connected at its outer end, through a special triplex reversing valve, either to the atmosphere or a chimney flue and (when lean-gas firing) to the lean-gas main in such a way that when odd-numbered oven regenerators are storing up heat from the combustion gases, even-numbered oven regenerators are preheating the air for combustion, or (when lean-gas firing) preheating the lean-gas and the air.

In cross-section each regenerator chamber is divided by partition walls into three sub-chambers, each of which communicates with a collecting flue connected to one branch of the triplex reversing valve at each face of the oven. For rich-gas heating all three sub-chambers are used for air regeneration, while for lean-gas heating the central sub-
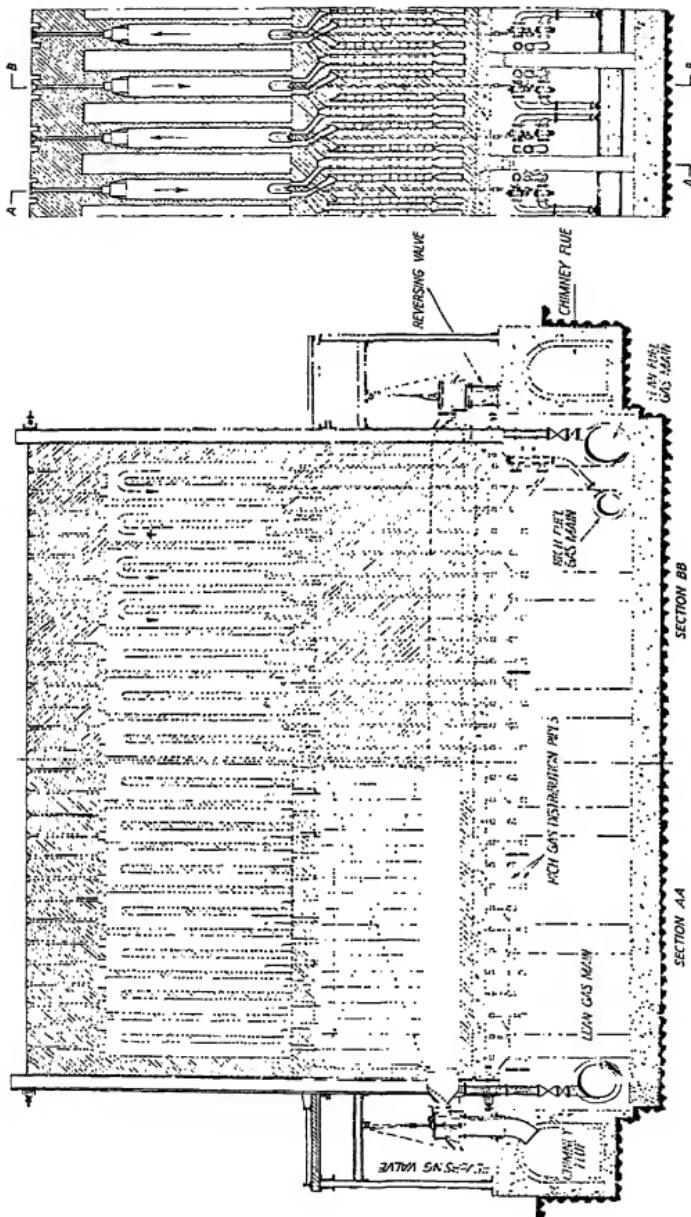
Fig. 8.—Simon-Carves "Underjet" Regenerative Compound Coke-oven of the Otto Twin-flue Design.

chambers preheat the gas and the two outer chambers preheat the air. When a regenerator is connected to the chimney all three sub-chambers, of course, pass out combustion gases.

The heating flues are of the twin or "hairpin" type. There are 28 vertical flues per wall, and during one heating, cycle combustion takes place in flues 1, 3, 5, 7, etc., flues 2, 4, 6, 8, etc. being exhaust flues. On reversal, the even-numbered flues become combustion flues and the odd-numbered exhaust flues. The two heating walls of one oven are staggered in this respect, odd flues in one wall and even flues in the other being combustion flues during the same period.

As the designation "Underjet" implies, the gas mains, burners and jets are located below the oven structure in a spacious basement formed by the reinforced concrete columns, beams and decking which support the oven brick-work. Along the sides of the basement run the chimney flues and gas mains carrying supplies of rich and lean fuel-gas. The lean-gas mains are connected at each oven to the triplex reversing valve referred to above. The rich-gas mains supply lateral pipes the full length of the oven heating wall, with a vertical burner pipe to each heating flue. In the burner pipes are fixed malleable iron jets, which are drilled out as required for calibration to 0·1 mm. to compensate for the variation in thickness of the coal charge due to the taper of the oven. The obvious advantage of this arrangement is that (the fuel-gas being distributed in metal pipes) an appreciable supply pressure can be used, which, maintained constant by a governor, ensures a definite column of gas passing to each burner as determined by the calibration of its jet. An initial calibration of the jets is all that is necessary, as any variation in the speed of coking is controlled simply by varying the gas pressure and draught. Should the throughput of the battery have at any time to be reduced so much that the supply pressure would be

dangerously low, loose wires are inserted in the jets to reduce the area by a definite amount.

Operation of Coke-ovens

Starting up a Modern Silica Coke-oven Battery.—In heating up a battery the essential point to remember is that silica material expands approximately 1·3 per cent. up to a temperature of about 600° C. Firebrick and silica should be carefully dried, so that there is no sudden evolution of steam in the pores of the bricks to cause spalling and cracking. Therefore, a considerable time should be taken in heating up the first 100° C. The most important thermal expansion of a well-burnt silica brick occurs between 150° and 250° C. After 250° C. there is no more sudden expansion of any appreciable magnitude except for a slight increase in volume due to unchanged quartz at about 575° C. All these expansions are reversible. The structure must, therefore, be heated very slowly up to 250° C., then a little faster up to 600° C., when the rate can be advanced rapidly.

Firstly, the ovens must be dried out and a very small amount of heat applied. The method of heating up will vary with circumstances. Should another battery be conveniently near, coke-oven gas may be used ; if there are gas producers near, producer gas may be utilised. Gas bought from gasworks may occasionally be used. Price and the economic situation will generally be the determining factors.

Should it be decided to utilise fires to heat up, usually two special bricks are built into the top of the oven-wall at each end, these bricks having round holes through them connecting the oven and flue. Very small wood fires are lighted at each end of the oven-chamber, on a temporary firebrick fireplace near the oven-door, and the smoke is drawn through the holes in the special bricks into the combustion flues, regenerators, waste-heat flues and thus to the chimney,

at the foot of which a fire is usually lighted at first to create the necessary draught.

By adjusting the dampers, the warm gases are distributed over all the flues and regenerators, and the drying out can proceed slowly. This may take about three weeks. For the first half, wood fires are used and then coke fires, which are gradually increased in size daily, thus slowly increasing the flue temperatures. For the first week it is advisable that the heating should not rise more than 5° C. per day. Temperatures should be taken every two or three hours and a schedule of temperatures, drawn up beforehand, strictly adhered to. Care should be taken to ensure even heating of the whole battery. The tie rods across the battery should have blocks of soft wood fitted inside each nut so that the wood can give a little as the silica expands. These nuts should be loosened regularly as required after the first ten days until the temperatures reach 700° C.

After 200° C. the fires are increased a little quicker. At about 204° C. a light yellow glow is seen in the flues during darkness, while at 400° C. the flues appear a reddish colour in the dark. At 480° C. they are red in the twilight, and at 600° C. they are red in the daylight. At 1000° C. they are a cherry red. The temporary brick-built fireplaces in the ovens are now pushed out of the ovens by a ram pusher, the round holes in the special bricks between oven-chamber and flue are filled up with a special round brick to fit, the doors are put on, and the ovens are then charged with coal —the temperatures having attained about 1000° C. The gases generated are allowed to run through the system and then bleed into the air for a short time before being led to the burners ; after testing, this bleeder is lit, and then the burners are lit under each oven.

There has been described a method of heating up ovens by oil firing. A small fire of coke is made on a false hearth, and a small oil flame is placed in the oven chamber so that the flame cannot impinge on the bricks, the walls being

protected by the false hearth. The oil fire must be main-
tained very small so that until 300° C. is reached the rate
of increase is only 10° C. per day. After this the rate of
heating can be increased to three or four times this amount
until 700° C. is reached. The ovens can then be brought
to full heat as rapidly as desired. The oil for firing is
contained in an overhead tank which feeds the burners.
Special oil burners are necessary, the air being blown in
under pressure. The temperature in the flues in every wall
should be measured at intervals every day, and great care
taken to ensure even heating of the whole structure. When
the temperature reaches 1000° C., the false hearths are
pushed out and the ovens charged with coal in the usual
manner. It has been said that this method may entail
expensive fittings of pipes and burners.

Another method is by utilising producer gas. Nowadays,
many types of regenerative ovens are built to burn either
producer or other low-grade gas alternatively with coke-oven
gas as desired. By using an external mechanical producer
the breeze can be used for heating the ovens. The producer
gas should be thoroughly cleaned before being taken to the
ovens. . Usually the gas from the producer is first passed
through a dust extractor, which removes the larger particles
of dust ; it is then taken to a washer cooler where it is
treated with fine sprays of water, which clean the last traces
of dust out of the producer gases.

Should the ovens be near a blast-furnace plant, blast-
furnace gas can be used for heating the ovens. This gas
should pass through a preheater close to the ovens in order
to maintain the gas at a constant temperature slightly in
excess of its dew-point, so that any deposition of water is
prevented. It is a well known fact that smooth and even
heating of an oven wall can be maintained when using a
gas of low calorific value, such as blast-furnace or producer
gas, more readily than when using a rich coke-oven gas or
coal-gas of as high as 570 B.T.U. per cubic foot. The one

essential in using producer gas is to have it thoroughly cleaned. Should there be another coke-oven battery in the near vicinity the easiest way is to heat up a new battery with coke-oven gas or coal-gas, piped from the adjoining cokeworks.

Whatever agent is used for heating up a coke-oven battery, the general principles are to dry out very slowly, to heat up slowly at a regular and steady rate, and to allow the expansions to take place slowly.

Maximum Economy in Heating Coke-ovens.—To attain a practical maximum of economy in the heating of a battery of coke-ovens it is necessary to observe the following points :—

(a) *Gas Supply.*—The supply of gas to each vertical or horizontal flue should suffer no obstruction by tar or other deposit in the aperture which determines the amount of gas passing to the flue. Each nozzle should pass sufficient gas to give even heating over the whole oven wall. A regular system of supervision and cleaning is essential to ensure a good gas distribution.

(b) *Air Supply.*—A flue-gas analysis at the chimney stack, followed by an analysis for each set of flues, will show the amount of excess air used. Abnormal coking times may often be attributed to excessive air supply and may be obviated by reducing the draught in a defective oven or by reducing the air port opening. Some types of ovens do not lend themselves to individual control, and adjustment of the main damper may be necessary, but adjustment of the air supply is important to give correct mixture of gas and air.

(c) *Draught.*—In those systems which adopt biscuit dampers to compensate for the varying draught

along a top horizontal flue, it should be seen that the setting of the dampers is correct.

Pushing Schedules.—In order to get the best yields and regular, smooth working, it is essential to push the ovens on a system, in order to keep regular coking times, and to ensure that an oven is not pushed if an adjoining one is empty, which would cause undue strain on the walls. The system of pushing by " threes " is a common method. For example, Nos. 1, 11, 21, 31, 41, 51 are pushed ; then 4, 14, 24, 34, 44, 54 ; then another three on, 7, 17, 27, 37, 47, 57 ; and so on. The ram-man should keep a list of the ovens pushed and the exact time of pushing and charging, a note being made for the management of the reason for any delay.

Repair and Relining of Ovens.—It is of advantage to a coke-oven manager to have available a reliable bricklayer who has had experience in building coke-oven walls. When any repairs are needed such a man is of much value. Silica walls, themselves, if they have been treated carefully, and the battery has not been shut down too often, should require very little attention for a great number of years. Should the joints alone leak, a spraying method has come to the fore, which in many cases appears to have given satisfaction. In the *C.O.M.A. Year Book* 1937 (344–348) is given a report of a discussion following a film on " Coke-oven Repairs by Cement Spraying." Finely ground special silica cement is mixed to a consistency of thin cream and sprayed through a fine jet on to the oven wall when hot, immediately after the coke has been discharged, and thus fills up open joints or cracks. In the discussion, Mr. Dinsdale described another method, in which he had let each oven down in turn to 100° C., pointed it with clay, put in new bricks where required, washed with cement and heated the ovens up again gradually. He took seven weeks to do an oven, from

letting down to starting again, and he said it was almost as right as when built.

Should much rebuilding be required, the new bricks or shapes must be dried carefully for a week beforehand, in some warm place. One or two ovens on each side of the wall to be rebuilt must be left empty and the temperature lowered slowly. When the wall itself is cool enough to allow working inside the oven, the old wall where necessary should be removed, the roof being carefully supported with timber, and then rebuilt with the dried new shapes. Before heating, the bricklayer should carefully point the walls and · give them a wash with cement mixture. The doors should then be replaced and the wall allowed to dry slowly with only the heat from the adjoining ovens, for about two days ; after this a very small flame may be allowed below the flues and the temperature permitted to rise slowly as when heating a new wall. Joints should be left for expansion, and if the joints between the shapes are made fairly large they will be found to close up on heating. Up to 250° C. the rise in temperature must be very slow, then quicker to 575° C., after which the flue temperatures can rapidly be brought to the usual values.

Should the whole length of the oven wall be rebuilt, it will be advisable to leave a space of at least 2 ins. between each end of the oven and the buckstay plates to allow for expansion, as normally the silica walls of the oven will expand in length approximately 4 to 5 ins. in all, that is to say, about 2 ins. at each end. The tie rods should be slackened to allow for this expansion and tightened up after the heating up is completed.

Modern Oven Machinery

Coal Charging Lorry.—The crushed coal is delivered into the coal charging lorry through chutes from the bottom of the service bunker. The standard type of coal lorry is

electrically propelled, and has four hoppers and the same number of charge holes fitted with mechanically operated agitating gear. These hoppers should be provided with poking holes accessible from the platform, and with telescopic cast iron drop sleeves or spouts leading from the hopper outlet to guide the coal into the charging holes of the oven and to prevent spillage during the charging. This charging car contains one full oven charge, which is automatically weighed before travelling by means of a weigh machine situated beneath the bunker. These cars are recommended to be mounted on four travelling wheels with sprung axle bearings. They travel at about 200 to 300 feet per minute along rails laid along the oven top, with a driving motor of approximately 30 H.P.

Combined Door Extractor, Coke Pusher and Coal Leveller Machine.—On modern ovens the large structural framework of this combined machine, weighing often 100 tons, is mounted on four to eight (preferably eight for large batteries) travelling wheels and moves along rails supported generally on a reinforced concrete raft. The door extractor mechanism may be one of two types :—it either lifts and draws back the plug-in self-sealing door, and the whole machine moves to one side to adjust the ram-head opposite the oven ; or the door may be first lifted slightly and then withdrawn and slued to one side clear of the path of the ram, thus enabling only one setting of the combined machine to be made. The ram-head on the front of the ram-beam (which often has a scraper fixed on the top, to descurf the carbon and prevent undue accumulation on the oven top) then pushes the coke out of the oven at a speed up to 90 feet per minute ; thus little more than a minute is needed to push and withdraw the ram-beam, and replace the doors. The ram-beam should be provided with a safety segment to prevent overrun. Sufficient platforms are provided to give easy access to all parts.

The Rope Operated Coal Levelling Bar should be fitted in a frame capable of vertical adjustment. Both the ram-beam and the leveller bar should have means of withdrawal by hand, in the event of the power failing. The machine carries also motors for the ram, the door lifter and the leveller, as well as a travelling motor. An operator's cab is provided, from which he can see in all directions, watch the ram-head going through the oven and at the same time is given some protection from the intense heat. Ammeters should be provided with these motors to prevent him from overloading, or damaging the machine or the oven walls.

A Combined Coke Guide and Door Extractor is on the opposite side of the oven to the ram. The machine comprises an undercarriage, equipped with travelling gear, which runs on rails on the coke side of the battery. The coke guide part of this machine consists of a frame of two parallel vertical grids of iron, which is run opposite the open oven to guide the hot coke into the quenching car, after the door has been removed by a door-lifting machine similar to that on the pusher machine.

CHAPTER V

COKE PRODUCTION

THE method of quenching and handling coke now generally preferred in modern practice is the discharging of an oven of coke into a coke car, which is then driven by an electric locomotive to a central quenching tower, where a measured quantity of water is dropped on to the coke from an overhead tank. The car is then drawn from the quenching station, allowed to steam off for a few minutes, and the coke is distributed on to a loading wharf, from which it is conveyed on a rubber belt to a screening plant. Here the coke is sized according to the market demand, the larger coke over 2 or 3 ins. being first extracted, possibly by a grizzley screen, and the smalls being then thoroughly screened and graded into suitable sizes for the market, possibly five :—
No. 1, $2\frac{5}{8}$ to 3 ins. ; No. 2, $1\frac{5}{8}$ to $2\frac{5}{8}$ ins. ; No. 3, $\frac{7}{8}$ to $1\frac{5}{8}$ ins. ; No. 4, $\frac{3}{8}$ to $\frac{7}{8}$ inch ; and smallest breeze.

Coke Quenching Car.—On all modern plants of over 3000 tons weekly capacity, it is now the practice to employ a coke quenching car, into which the red hot coke is pushed through the coke guide. This car is arranged with a sloping bottom, really functioning as a moving inclined bench. While the coke is being discharged, the car is moving along slowly in order to distribute the coke uniformly over the car bottom. An electric locomotive then takes the car and its contents to a central quenching station, where it is deluged for a specified time by a measured quantity of water. The steam is thus taken away from the ovens so

that the brickwork is not damaged.   Approximately 6000 gallons of water are sprayed on to the coke in some 45 seconds ;   the amount can be regulated to ensure proper quenching and that no undue water is used.   After the water has been turned off, the coke is allowed to remain on the car for about one minute to steam and allow the water to drain off.   About one-sixth of the water evaporates.   The drainage runs into a tank where the fine coke settles out, and may be removed periodically with a grab.   The water can then be pumped up to an overhead tank for further use.   The coke is then taken by the coke car to a coke wharf, large enough to hold the contents of three or four ovens, and discharged thereon.   Along the coke wharf are arranged small quenching hoses for spot quenching.   When the coke has cooled sufficiently, finger gates at the lower end of the coke bench are raised, thereby allowing the coke to fall on to a moving rubber belt conveyor, which carries the coke to the screening plant.

Transport of Coal and Coke : Belt Conveyors.—Coal is usually brought to the coke-oven plant by rail in railway trucks and coke is usually despatched by the same means. However, in transporting coal from the tipping well to the blending bunker and from the blending bunker to the crusher, it is now usual to make use of rubber belt conveyors.   These are also used in conveying the coke from the bench to the screening plant, and from screen to screen ; finally, the large coke may be carried by the belt conveyor to the blast-furnace plant, although generally the different sizes of coke are lowered from the screens into railway trucks for transportation to their several destinations.   Generally, the crushed coal is elevated up into the storage bunker by bucket elevators of steel or malleable iron, if the incline is almost vertical, but if it is a long, slight incline a rubber belt conveyor may be used.

These conveyors are endless belts running about end

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pulleys and supported on the runs by a series of securely mounted idlers, usually of the trough variety on the conveying run and flat on the return run, though " special purpose " belt conveyors are sometimes furnished with straight or flat idlers on the carrying run. The individual pulleys comprising the trough type idlers are of uniform diameter, so that the peripheral speed of the pulleys at all points of contact with the travelling belt is the same as the belt speed. Troughing idlers may consist of any number of individual pulleys, but an odd number is to be preferred, in order that the centre section of the conveying belt may be properly supported. Return idlers, supporting the return run of the belt, are flat and may be made up of a set of individual pulleys mounted on a common shaft or a single pulley slightly wider than the width of the belt it supports. These return idlers are spaced at about 10-ft. intervals, while the troughing and load-carrying idlers are spaced from 2 to 5 ft. apart, depending upon the width of the belt and the weight of material—the wider the belt, the closer are the idlers spaced. At all loading points, the troughing idlers should be spaced at about two-thirds the spacing required on conveying stretches of the belt and a troughing idler should be spaced at about 6 ins. to the rear of the discharging lip of the loading chute to give added support to the belt. Where the carrying belt takes an abrupt change in direction, a straight-face bend pulley is substituted for a troughing idler at the point at which the bend occurs and the troughing idlers immediately preceding and following the bend pulley should be situated from it at a distance somewhat less than the spacing of the idlers on straight carrying sections of the conveyor. When belt conveyors exceed 25 ft. in length (they are built to 1000 ft. in length) guide idlers mounted normally to the sides of the belt on the carrying side should be installed about every 25 ft. to prevent the belt from leaving the troughing idlers.

Discharge of load occurs over the head-end pulley of the

conveyor, or may take place at any point along the conveyor, if the carrying belt is carried over an elevated discharge pulley and then around a lower bend pulley (straight) situated slightly to the rear of the discharge pulley so that the carrying belt takes a reverse S loop. Such discharge points may be permanently located, in which case they are called "fixed dumps," or the discharge and bend pulleys may be mounted on a travelling carriage and shifted from place to place on a horizontal section of the conveyor, in which case the discharge carriage is called a "tripper." Loading and discharging belt conveyors should be through the agency of chutes so inclined that the velocity of the material leaving the chute is as nearly as possible the same as the speed of the conveyor belt, and in the same direction.

The driving pulley, which should be heavily crowned in order to maintain alignment of the belt, is advisably the head pulley of the conveyor, in order that the tension side of the belt may carry the load, but when this arrangement is not feasible the drive may be located at the tail end of the conveyor, or at any point along its lower run by looping the return belt much as the carrying belt is looped for a discharge point. For unusually long belt conveyors, two driving pulleys geared together are employed, in which case the driving section of the belt forms a double loop. A still more powerful drive is secured by covering the driving pulleys with lagging to increase their grip on the conveyor belt. Owing to the unavoidable stretch of a conveyor belt, particularly when first installed and operated, take-ups with generous adjustment should be provided, customarily at the end of the conveyor removed from the drive. Take-ups can also be installed on the return belt on a loop similar to that required for centre drives.

If the conveyor is to be employed handling damp coal, which has a tendency to adhere to the belt, rotary brushes should be installed at the head end of the conveyor and at the discharge pulleys of fixed dumps or trippers. The brush

should be set against the underside of the belt a little to the rear of the conveyor pulley, so that the trajectory of the sweepings will cause them to land on the discharging chutes.

Loading chutes should not only be of generous pitch to secure the desirable speed of the material flow, but should discharge to the conveyor belt over a curved lip, so as to minimise the shock of impact, and should have guiding skirt boards for a distance of from two to three troughing idlers to allow the load to settle and adjust itself on the belt before it passes the confines of the skirt boards. Triangular cleats should run the full length of the skirt boards to assist in properly distributing the load, and the distance between the skirt boards should be several inches less than the width of the conveyor belt, depending upon the width of the belt.

The speeds of belt conveyors vary greatly, from 50 feet per minute to 400 feet per minute. Heavy material can, as a rule, be conveyed at a higher speed than light material. Belt conveyors are limited in their ability to carry loads up an incline of more than 20°, and have to be erected in one vertical plane, *i.e.* they cannot turn corners—though a series of conveyors can discharge from one to another, each running in a different direction. Coke, with an average weight of 33·5 lbs. per cubic foot, has an economic belt speed of about 250 feet per minute, while fine coal, being heavier, about 50 lbs. per cubic foot, has a greater economic speed, of approximately 400 feet per minute. The width of the belt conveyor affects the amount that can be conveyed, and the capacity in tons per hour can be calculated by the following equation :

$$\text{Capacity in tons per hour} = W = \frac{Kw^2Vd}{100,000},$$

where K is a constant, its value being 1·5 ;

w = width of belt in inches ;

V = speed of conveyor in feet per minute ;

d = density of material in lbs. per cubic foot.

Power Requirements.—The consumption of power by belt conveyors is very small, and depends somewhat upon the method of lubrication. It is so low that the power requirements of a well greased conveyor may be standardised according to the following formula :

$$\text{H.P.} = \frac{(0.00009w^2V + 0.08W)L + WH}{1,000},$$

where w = width of belt in inches ;

V = speed of conveyor in feet per minute ;

W = load in tons per hour ;

L = total length of conveyor in feet ;

H = rise in length of conveyor in feet.

The total expense of operating a belt conveyor system is best expressed as cost per ton of material handled.

W. Scholes (*C.O.M.A. Year Book* 1934, 137) states that with a belt 36 ins. wide and 300 ft. long, at a speed of 218 ft. per min., a tonnage of 1,500,000 has been obtained. These large coke-wharf belts had $\frac{3}{16}$-inch rubber cover on the carrying side and $\frac{1}{16}$-inch rubber on the underside. The number and weight of plies in a belt are a function of its size and power requirements. A layer of open-woven fabric embedded in the top cover may act as a reinforcement.

Screening.—On a modern coking plant good coke screening is most essential ; in the first instance to remove all the dust or smalls from furnace or foundry coke, and secondly to ensure that the smaller nuts are well screened and correctly graded to compete successfully in the ever growing domestic coke market.

There are a number of methods of screening, but the most efficient method is one where the largest sizes are removed first. For separating the large and small coke, the grizzley type of screen is perhaps the most popular. Its pre-eminence can be accounted for by its robustness in handling big tonnages, simplicity of construction and low running

cost. W. Scholes (*C.O.M.A. Year Book* 1934, 139) states that the screening cost for grizzleys on two plants handling 50 and 75 tons per hour was a shade under 0·1*d*. and that there was no appreciable difference in the cost of replacements, whether using cast iron or manganese steel discs. Manganese steel discs were, however, recommended because, although they were more expensive, the work entailed in renewal was not so frequent, and also there was very little danger of pieces breaking off and causing damage to any coke cutter interposed in the system.

Grizzley Screens are composed of a series of shafts placed transversely to the flow of the material, and fitted with either plain or serrated discs. The space between the discs forms the screening apertures. The rotation of the discs propels and disturbs the material, and they are used for rough separation or elimination of small.

For the grading of the different sizes of small, some types of flat vibratory screens are rapidly replacing all other screens. H. O. H. Cerckel (*C.O.M.A. Year Book* 1938, 183) states that vibrators are easy to instal and drive, take up little room and, being light, can have cheap supporting structures. The method of producing the vibrations is by revolving an unbalanced pulley or shaft, by make and break contacts, etc. The best results are obtained when the screen itself is secured in a frame which is vibrated bodily. When screening $\frac{3}{4}$-inch material or above, the speed of travel can be increased by putting the screen to a steeper angle.

The Vibrex is a well-known type which has vibration imparted to it by an unbalanced wheel running at about 2000 r.p.m., the frame being floated on springs secured to the undercarriage. An important point is the tensioning of the wire mesh, the Vibrex and also the Gyrex types having a camber frame which renders tensioning a simple matter. The slight camber given to the mesh also serves to distribute the bed of coke evenly through the transverse

screen. In dead flat types of screens, the mat must be tensioned in a special frame, the complete assembly afterwards being mounted on the screen frame.

When perforated plates are used for screening, only about 40 per cent. is open screening area ; they do not give as good results as wire screens or bar grate types. Step-shaped perforated plates are more efficient, as each step disturbs the material, bringing the smalls in contact with the plates, where they are quickly eliminated by the staggering of the holes. Vibratory screen wire mats made from 0·5 per cent. carbon steel with doubly crimped mesh are said to give good service. The power requirements of modern flat screens are small, ranging from $\frac{1}{2}$ H.P. for the smallest to 5 H.P. for the largest standard make.

Rotary type screens are not very efficient, as, to be so, they must be well above their work, with a comparatively small bed of coke passing through. The removal of the smallest sizes first is not good.

An efficient screening plant usually removes the largest sizes first, the smaller then dropping lower to another screen which may remove, say, the $1\frac{5}{8}$-inch to 2-inch or $2\frac{5}{8}$-inch, depending on what size is removed from the large furnace coke ; then another vibrating screen removes, say, the $\frac{7}{8}$-inch to $1\frac{5}{8}$-inch, another the $\frac{5}{8}$-inch, and that falling through is the $\frac{5}{8}$-inch to 0-inch or ballast. Possibly some arrangement of rubber belt conveyors may be installed, conveying the sizes away, or to another screen as necessary. It will entirely depend on the market demand what different sizes are removed. Whichever sizes are made, it is essential that they are true to size, well graded and free from dust. The quality should be of the highest and improvement always aimed at, to capture—to satisfy—and to keep an ever increasing domestic market.

Coke Cutting.—When the market for large coke is poor, it is sometimes advisable to cut the coke into nut sizes for

household and central heating sales. Two types of coke cutters on the market are the " Cortes " and the " Nelson." They have a normal capacity of up to 35 tons per hour with hard coke, but with a fairly soft coke they can deal with 50 tons per hour. Approximately 5 per cent. ballast is made in the process of cutting nuts.

Blast-furnace Coke is the principal product of most coke-oven plants. The best coke for this purpose should have :—

(1) *Mechanical strength,* sufficient to withstand the crushing strain in the blast-furnace.

(2) *Size over 2 ins.,* and graded if possible, with freedom from breeze.

(3) *Hardness* or resistance to impact, a lack of brittleness permitting rough handling without undue fragmentation.

(4) *Porosity,* in order that the coke may expose a maximum surface for reaction with the furnace gases (CO_2).

(5) *Low ash content,* under 8 per cent. if possible.

(6) *Low moisture,* under 1 per cent.

(7) *Low phosphorus,* under 0·02 per cent.

(8) *Low sulphur,* under 1·25 per cent.

(9) *High carbon content.*

Blast-furnace coke makers generally require a uniform size, with an absence of breeze and dust the most important points. It is now usually wanted over 2 ins., and graded rather than very large. In America and Germany they prefer it from 3 to 4 ins. rather than from 2 to 6 ins. In this country they are gradually coming to the same opinion. A regular size, so that the blast has not to be altered, seems to be what is required. R. V. Wheeler and R. A. Mott advocate $1\frac{1}{2}$ in. as the minimum size. One must remember that in transport there is always liable to be some breakage,

therefore a tough coke which does not break much after screening is to be aimed at.

In testing coke for blast-furnace use, firstly the usual chemical tests for ash, moisture, phosphorus, sulphur and carbon content are applied. If these are satisfactory, size and hardness are then examined. The standard *shatter test* is universally used and is considered to be an index of the suitability of coke for blast-furnace use. The Midland, Northern and Scottish Coke Research Committees have adopted and advocate the following standard method for the shatter test :—

(1) *Apparatus.*—The shatter test apparatus shall consist of a box 18 ins. in width, 28 ins. in length and approximately 15 ins. in depth, supported above a rigidly mounted cast iron or steel plate, not less than $\frac{1}{2}$ inch in thickness, and not less than 38 ins. in width by 48 ins. in length. The inside of the bottom of the box when in its highest position shall be 6 ft. above the plate. The bottom of the box shall consist of two doors hinged lengthwise and latched so that they will swing open freely and not impede the fall of the coke. Boards about 8 ins. in height shall be placed around the plate so that no coke is lost. To minimise the breakage of coke which might otherwise occur while placing the sample in the box, the box shall be so suspended that it can be lowered to a convenient level for filling.

(2) *Screens.*—For determining the breakage of the coke sample, square mesh screens with the following openings shall be used :—2 ins., $1\frac{1}{2}$ ins., 1 inch and $\frac{1}{2}$ inch.

(3) *Procedure.*—Fifty pounds of coke, all over 2 ins., shall be placed in the box of the shatter test apparatus, the coke levelled, the box raised and the coke dropped on to the plate. The box shall be lowered and all the coke shovelled into it indiscriminately, but taking care to avoid breakage. The box shall then be raised and the box again dropped. When the coke has been dropped thus four times in all, it shall be screened by hand in such a way that any piece that can pass through the screens in any position shall be counted as undersize, and the weights of coke of each grade shall be determined to the nearest ounce.

(4) *Statement of Results.*—At least three tests shall be made and the

results stated as percentages, to one decimal place, remaining on the 2 ins., 1½ ins., 1 inch and ½ inch screens. The shatter indices reported shall be the average values of the percentages retained on the 2 ins. and the 1½ ins. screens, and shall be given to the nearest whole number only.

A single figure shall be understood to be the 2 ins. index unless otherwise stated. In future the 1½ ins. figure shall invariably be given in addition to the 2 ins. figure by stating the shatter indices in the form 74/86, where the figures are the 2 ins. and 1½ ins. indices respectively.

Where possible the statement of the shatter indices shall be accompanied by a brief description of the coke ; for example : "blocky coke," "prismatic coke," "beehive coke."

Another test that is coming more to the fore is the *abrasive* or *Trummel test*, which is carried out by rotating about 30 lbs. of coke in a barrel. The Micum Trummel test is the standard German test for the mechanical strength of coke. An average sample of the coke to be tested is taken with a fork having prongs 50 mm., or approximately 2 ins., apart. 50 kilograms of the sample is placed in a cylindrical steel drum 1 metre long and 1 metre in diameter, which can be rotated mechanically in a horizontal plane. Four angle-irons projecting 100 mm. are fixed to the inside walls of the drum at regular intervals of 90°, and the drum is geared to make 100 revolutions in 4 minutes ($\pm$ 10 seconds). After 100 revolutions the contents are sieved by shaking over a series of round-holed sieves (diameters 100, 80, 60, 40, 20 and 10 mm.). The percentage remaining on the 40 mm. round-holed sieve is considered as the Trummel index, and the measure of the mechanical strength of that coke, while attention is also given to the percentage passing the 100 mm. sieve as an index of abradibility.

The above two tests are the chief means of measuring the strength of blast-furnace coke.

It is generally considered that a porosity of 50 to 52 per cent. is a suitable figure for blast-furnace coke.

Ash in coke involves the use of lime, in the blast-furnace, to form a slag, and additional quantities of coke to flux the

slag, so that large increases in coke consumption are needed with excessive ash. Whether coke should be combustible or not is a debatable point.

Foundry Coke.—In foundry practice the burden to be carried is different from in a blast-furnace, and no reducing action is necessary, the function of the coke being purely thermal. Foundry practice demands a coke of high calorific value and hardness for the burden and severe abrasion it receives with the pig-iron used. In practice a dense, non-fingery, well burnt, fairly large coke is considered necessary, while the importance of ash, sulphur, phosphorus and water content is strongly stressed. Foundry coke should have an ash content of below 8 per cent.; the sulphur content should be less than 1·0 per cent., and preferably below 0·8 per cent.; phosphorus, below 0·02 per cent.; while its water content should be very low because of its carbon-diluting effect. The shatter index figure should be over 90 on 1½ ins.

The main requirements of a good foundry coke are that it should have the property of melting the maximum weight of metal with a minimum consumption of fuel, and have the ability to produce a high combustion temperature.

Central Heating and Household Coke.—After removing the larger coke over, say, 3 ins. at the screening plant, the smaller sizes are carefully screened and graded. These special-size coke nuts, graded to proper sizes, are sold for use in steam boilers, domestic boilers, central heating boilers, Smith's hearths, small greenhouse boilers, domestic open grates and for many other purposes. For all these the most important point is to get the nuts properly sized.

The National Smoke Abatement Society are constantly stressing the damage done by smoke and urging the use of smokeless fuel. The domestic smoke problem is still to be tackled, and the use of coke nuts in many boilers and special

open grates was a step in the right direction. In recent years considerable attention has been paid to the design of special fire grates to burn coke in an open fire for room heating. High temperature coke cannot easily be burnt in a well grate ; bars are required and these need wider spacing than in a coal fire to enable a better current of air to reach the fuel. A number of grates are now on the market, many of which are provided with gas jets or gas pokers to light the fires. The lighting of oven coke was a slow process without some such aid, as in most oven coke there was only about 1 to $2\frac{1}{2}$ per cent. of volatile matter ; possibly if some 5 to 6 per cent. of volatile matter could be left in coke for household use it would be an improvement. As regards cost, R. A. Mott has stated (*C.O.M.A. Year Book* 1933, 309) that, when the outside temperature was 50° F., a coke-fired boiler and oven would use 25 lbs. of coke per day, which at 30*s.* per ton would cost 4*d.* per day, and this should meet the needs of those living in small houses.

The main points to consider in coke for use in household open fires are :—

(1) Ignitibility ; it is important that it should be free-burning and easily ignited.

(2) Uniformity of size ; it must be carefully graded ; $1\frac{1}{2}$-inch nuts are generally considered best for open fires.

(3) Combustibility.

(4) Ash content to be low.

(5) Ash volume to be low.

(6) Reactivity to carbon dioxide.

(7) Bulk density.

It is estimated that domestic and general industrial coke consumption is approximately 13 million tons a year (9 million tons gas coke and 4 million tons coke-oven coke). The demand is increasing in the London area, where there are now about 150,000 domestic coke boilers installed each

year, consuming 450,000 tons of coke. Power stations are taking 250,000 tons of breeze, and the open coke fire sales have reached 100,000 tons a year in the area covered by the London and Counties Coke Association. Thus, more and more domestic graded small coke is going into the market. The coke-oven owners must keep improving their product to keep and increase this growing market. Every possible outlet for the sale of coke should be considered and new outlets investigated. Below are two new prospects :—

(1) *Coke for Grass Drying Plants*.—During the past three years agriculture has become an increasing consumer of coke for grass drying plants. At the present time there are some 100 plants in operation, each consuming up to 200 tons of coke per annum, generally during the summer months. A producer type of furnace is employed now on most of these plants, and the fuel cost per ton of dried grass has been brought down to 18s. 10d.

(2) *Soil Warming*.—The Hertfordshire Institute of Agriculture has developed soil warming by means of hot water pipes in tomato houses, and they state that the initial cost was more than repaid during the first year by the increased crops (W. L. Boon and G. E. Foxwell, *Gas World*, **109**, 1938 —*Coking Section*, 10).

CHAPTER VI

TREATMENT AND TESTING OF GAS

THE function of the whole of the plant between the ovens and the primary coolers is :—(1) to conduct the gases without obstruction from the ovens to the by-product plant ; (2) to reduce the heat content of the gas by atmospheric cooling as far as possible ; and (3) to condense and remove the heavier tar vapour and ammonium chloride fumes which might otherwise block up the tubes of the coolers.

The plant consists of the following :—the ascension pipe, the connecting piece between the ascension pipe and the gas collecting main, the valve, the collecting main and its attendant liquor circulating devices, and, at the end of the collecting main, the tar trap.

It is customary in modern plants to line the ascension pipe with bricks, and they are kept so hot that no condensation can take place, which prevents distillation of tar which would otherwise condense on the inner face of the ascension pipes. In the valve piece connecting the ascension pipes with the collecting main is the tar liquor spray, and this cools the gas rapidly and at the same time keeps the valve clean. The provision of a spray at each ascension pipe permits such a large volume of cool tar liquor to be introduced the whole time that the collecting main is kept comparatively cool, and the hot gases can produce no distillation of the tar, which used to be the cause of pitch residue being formed on the bottom of the main ; thus the cleaning of the mains is almost done away with in modern coke-ovens.

Double Collecting Mains.—Since the oven-doors on modern coke-ovens are not lifted up by a winch on the top, but are withdrawn by machines from the end, the top of the ovens can on both sides be occupied by collecting mains. While, as a rule, one collecting main suffices for each battery, double collecting mains are of advantage on plants carbonising a highly volatile coal at high temperatures, so as to relieve the chambers from high gas pressures, which cause losses by diffusion through the walls and by decomposition of the hydrocarbons.

In the older plants, the temperature of the gases in the collecting main was normally 300° to 400° C., but the installation of liquor sprays in modern ovens reduces the temperature of the gas to 80° or 100° C.

In plants where the valves are not liquor sprayed, liquor or tar is circulated through the mains by a special circulating pump for cleaning purposes, and, in addition, the mains require raking out daily in order that no deposits of pitch should form. A purpose of liquor circulation, whether in the form of sprays or in a stream along the bottom of the main, is to assist in the deposition of ammonium chloride, so that corrosion in later parts of the plant is avoided.

The tank, at the base of the conducting main, is raked out daily to remove any pitch that may have accumulated. With the circulation of tar and liquor, the temperature of the gases is reduced, but comparatively little heat is abstracted from the gas owing to the amount of liquor evaporated in the process. Unless liquor sprays on the valves are used as on modern plants, the operator cannot control the temperature of the gas before the coolers, and is more or less at the mercy of the atmosphere and of conditions in the ovens. In some modern plants, steam jackets are placed around the ascension pipes in order to generate high pressure steam.

Condensation or Cooling

The object of condensation is twofold :—(1) to cool the gases to a suitable temperature for scrubbing ; and (2) to eliminate tar and water vapour, and to remove ammoniacal liquor and a portion of the sulphuretted hydrogen.   Coolers in common use may be divided into two classes :—(a) atmospheric, (b) water.

The amount of condensing surface required depends upon the amount of condensable products deposited by the gas, and this varies with the class of coal. *Atmospheric condensers* require approximately 5 square feet of superficial cooling surface per thousand cubic feet of gas made per maximum day.   This includes the area of all piping between the collecting main and the outlet of the coolers. *Water cooled condensers* require approximately 3 square feet of water cooled surface per thousand cubic feet of gas passing per maximum day.

Atmospheric Coolers.—This form of condenser consists of a series of pipes exposed to the cooling effect of the air, and its action consists in the transmission of heat from the hot gases and vapours in the interior of the pipes to the external air in contact with the outer surfaces of the pipes ; this air, expanding, ascends by reason of its lower density, and is constantly being replaced by fresh air at a lower temperature and having a higher density.   The following are the most common kinds of atmospheric coolers :—(1) horizontal, (2) vertical, (3) annular, and (4) battery type.

(1) *The Horizontal Condenser or Cooler* consists of a series of pipes arranged side by side in pairs, the end of each length being joined to that of the next.   The pipes are supported on a framework, so arranged that, from the inlet at the top through the entire run of the apparatus to the outlet at the bottom, there is a gradual inclination, which enables the products of condensation to flow away to the

outlet (which is provided with a pipe dipping into a seal-pot) and thence to the tar well. Blank flanges are on the end of each length of pipe for steaming out as required.

(2) *The Vertical Cooler* consists of a series of pipes, usually 9-ft. lengths, placed vertically, through which the gases pass up and down alternately. This is brought about by attaching the pipes at the bottom to a cast iron box provided with a series of mid-feathers which dip to a certain depth in the liquids deposited from the condenser so as to form a seal. The pipes are connected by semicircular bends at the top. The condensed products flow from the collecting cistern through a seal-pot to the tar well.

(3) *The Annular Cooler* (often used as an oil cooler) consists of large pipes placed horizontally or vertically, each of which encloses a smaller pipe, thus forming an annular space through which the gas travels. In addition, there are other pipes placed diagonally, which connect alternately the tops and bottoms of the pipes. This arrangement has the effect of always causing the gas to travel through the annular space in a downward direction ; and the current of heated air rises upward through the inner air pipe. In addition, water may be sprayed on to the top of the bank of pipes as a second cooling medium.

(4) *The Battery Cooler or Condenser* consists of an oblong cast iron vessel 12 to 48 ins. wide, 12 to 24 ft. high, and of varying length up to 50 ft. It is divided internally by plates or mid-feathers extending to within about 12 ins. of the top and bottom of the apparatus alternately, so that the gas is compelled to pass from the inlet up and down to the outlet. In order to increase its condensing power, numerous 2-inch or 3-inch tubes are fitted, open at both ends so the air may pass through the vessel from side to side. Seal-pots are also provided.

Water Coolers are of two main types, namely, water-tube and multi-tubular. In the first of these, the hot gases

pass through the space around the tubes ; in the second
type the water passes around the tubes and the gas through
them. Several kinds of water cooled condensers are in use
at the present day, in many instances differing only in
detail. The older forms are usually composed of cast iron
plates bolted together, frequently with cast iron tubes
through which the gas passes. The modern type is usually
cylindrical, although it may be rectangular, and is con-
structed from mild steel or drawn steel plates. Preferably,
the water should pass through the tubes, which may be
from $2\frac{1}{2}$ to $4\frac{1}{2}$ inches bore, the gas circulating in the space
around the tubes. The height of the main shell should be
from two to five times the diameter, and the standard 20-ft.
tubes answer well. As regards the spacing of the tubes,
the free space in the shell should not be too greatly encum-
bered, otherwise stoppages will occur. It has been recom-
mended that the tubes occupy 18 per cent. of the total
area of the tube plates.

As regards the advantages of the respective types of con-
denser, advocates of the atmospheric type point out that
this apparatus relieves the gas from all sudden shock of
cooling, which tends to deposit some of the light-giving
hydrocarbons which might otherwise remain in the gas and
be useful, especially if the gas is sold for town supply. In
a water condenser, arrangements may be made so that the
flow of both gas and water is reversible. Advantages
claimed for the water cooled condensers are that the action
of the apparatus is more under control, and the greater
efficiency of water compared with air as a cooling medium.
All coolers should be fitted with suitable by-pass valves and
connections ; thermometers should be fitted on the inlet
and outlet pipes so that a check of the cooling effect may
be observed by those responsible. In very hot weather it
is advantageous to let a stream of water trickle over the
pipes of an air cooler, since the resulting evaporation induces
a certain amount of cooling.

Scrubbing

Tower Scrubbers.—The tower scrubber is usually a circular cast iron vessel built up in sections. It is anything from 7 to 20 feet in diameter and from 40 to 120 feet high. The vessel itself is simply a shell containing the scrubbing material, whose object is to break up the gas into small streams, and to produce as large a surface as possible, which is constantly wetted by means of the liquid run or sprayed in at the top. The material which is placed inside the scrubber is commonly composed of wooden boards, about $\frac{1}{2}$ inch thick and 4 to 8 inches deep, with $\frac{1}{2}$-inch distance pieces to keep them apart. These boards are arranged on edge in rows so that they assume a spiral form, and at certain distances one or two rows are left out in order to allow the finely divided streams of gas to unite, afterwards breaking up again to pass through another series of boards. The gas enters each scrubber at the bottom, meets the scrubbing liquor intimately while ascending and leaves at the top. Scrubbers are usually built in threes, the fresh scrubbing liquor meeting the gas in the third scrubber, and the fresh gas as it enters the first scrubber meeting the partly saturated liquor which has been through the other two scrubbers. In ammonia scrubbers, water is the scrubbing medium ; while in benzole scrubbers, some form of oil is used.

The disadvantages of tower scrubbers are :—

(1) Their liability to become choked or blocked, thus causing the stream of gas to ride up channels without coming into intimate contact with the descending liquid.

(2) They necessitate a fairly large supply of washing liquid and with ammonia use a large amount of water.

The advantages of tower scrubbers are :—

(1) Small ground space.

(2) Simplicity of construction.

(3) Low motive power required to supply the liquid to the top.

Rotary Scrubbers.—A common type of rotary scrubber consists of a rectangular tank, divided internally by cast iron plates into compartments which are filled with liquid. The compartments are so arranged that the liquor supplied at the gas outlet end is capable of flowing from compartment to compartment until it emerges at the gas inlet end. There is a shaft running the length of the apparatus, and attached to this are various devices for exposing a large amount of wetting surface to the action of the gas. The material used is generally wooden boards arranged in the form of bundles with distance pieces between, or circular brushes. As they slowly revolve with the motion of the shaft, they are completely wetted in every direction ; and as they rise from the liquid in the compartment, the crude gas passing through or amongst them comes into contact with the wet surfaces and is deprived of its ammonia and other impurities. Clean water enters at the gas outlet end and, flowing through the different compartments, meets the crude gas at the opposite end, gradually increasing in strength as it travels through the apparatus. The efficiency of rotary scrubbers largely depends on there being no slipping of the gas between each compartment, and this depends on the face joint between the division plates of each compartment, and the revolving collar attached to the shaft, being properly gas-tight.

Exhausters

The objects of the exhausters are :—(1) to overcome the resistance offered to the passage of the gas through the different parts of the by-product plant ; and (2) to effect the withdrawal of gas, as quickly as it is generated, from contact with the hot walls of the oven. The main advantage

of a good exhauster is that control can be kept over the quality of the gas made. The essential features of a good exhauster are that it should be simple in construction, work with a minimum of friction and power, and give a steady gauge, and that the internal parts should be as gas-tight as possible.

In modern plants the turbo high speed type is most generally used, though the low speed positive type, such as the Roots or Connersville, or the Beale exhauster may be used.

(1) **The Turbo or Centrifugal Exhauster** is being largely used on new plants, especially on those having direct ammonia plants. Owing to its high velocity, often 6,000 to 7,000 r.p.m., the dimensions are considerably reduced. They have the advantage of requiring little space, being driven by direct-connected electric motor or steam turbine. The turbo exhauster is operated on the principle of the centrifugal pump, having impellers fixed to a shaft which passes through a cast iron outer case, these impellers being rotated at high speed by a turbine ; their chief merit is that they avoid the rubbing contact and frictional losses inevitably associated with the ordinary blade type. A further advantage of the turbo exhauster is that it acts as a tar extractor, its high speed agglomerating the tar particles against the outer casing.

(2) **The Roots or Connersville Type** is an efficient and reliable exhauster over a wide range of capacities. It is generally driven by a direct-connected steam engine. It occupies more space than the turbo type. In this exhauster two hollow impellers of cast iron or cast steel revolve simultaneously in opposite directions in a casing, synchronism being secured by spur gearing. The impellers are of carefully designed type, the tip of each impeller engaging in the indented portion of the other impeller in such a way as to

avoid undue slip. The Carnegie Steel Co. of the U.S.A. use a number of these exhausters, each having a capacity of 40 million cubic feet of gas per 24 hours at 100 r.p.m.

(3) **The Beale Exhauster** occupies more space than the turbo exhauster. It consists of an outer case of an elliptical shape (Fig. 9), the width being greater than the height. There is an inner drum fitted with blades which are joined by a cast iron guide sliding on a rotating block fixed in the centre of the exhauster, where the velocity is lowest, and consequently a considerable reduction in friction is effected. The slides are drawn in and out as the drum revolves eccentrically to the outer case ; the blades have slips on springs touching the walls of the outer case and sweeping them continuously, resulting in the gas being sucked in at the inlet, carried round above the drum and discharged at the outlet.

Gauges should be provided in the exhauster house showing the suction or pressure at various points in the system, such as :—(a) the outlet of the gas collecting main, (b) inlet and outlet of each cooler, (c) before and after exhauster, (d) before and after tar extractor, (e) before and after each scrubber. These show the pressure from the ovens, which, with a knowledge of the depth of seal-pots on the various dip pipes, should give ample warning of any obstruction which may cause a seal to blow.

The most important point is efficient lubrication of the exhauster with the best oil recommended for the purpose. Mechanical lubricators should be fixed on all moving parts of the exhauster and its engine, and these must be kept filled to the proper level with the correct oil. The makers' instructions as to the oiling should be rigorously carried out. The correct grade of oil should be used and, to avoid contamination by dirt and the use of the wrong grade, bottles should be supplied with chained stoppers, and tins with
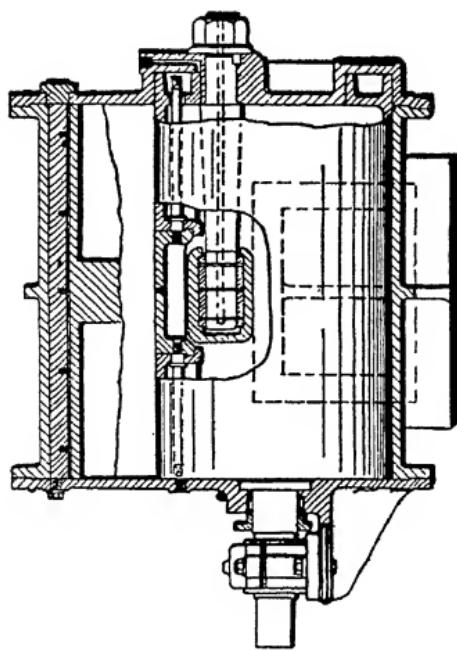
Fig. 9.—Beale Exhauster (the Bryan Donkin Co.).
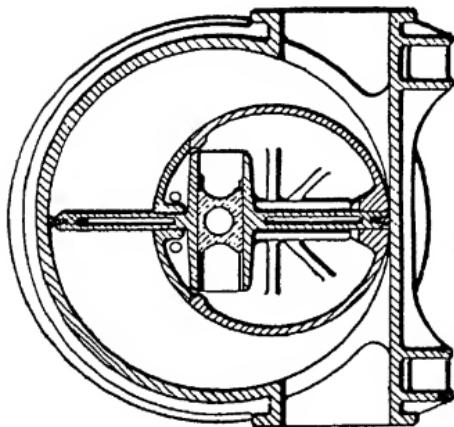
lids, each labelled with the name of the lubricant and the particular unit it is intended for. It can be truly said that the exhauster is the life of a coke and by-products plant.

Oxide Gas Purifiers

As gas sold sometimes has to be free from hydrogen sulphide, *i.e.* the gas has to pass the Gas Referees' lead acetate test, which is capable of detecting one part of hydrogen sulphide per million, and the coke-oven gas contains over 600 grains per 100 cu. ft., it may be necessary to instal some process of purification. The most common type, and that needing the least supervision, is oxide purification. This consists essentially in passing the gas through layers of moist oxide of iron, the following reactions taking place :—

$$Fe_2O_3.H_2O + 3H_2S \rightarrow Fe_2S_3 + 4H_2O ;$$
$$Fe_2O_3.H_2O + 3H_2S \rightarrow 2FeS + S + 4H_2O.$$
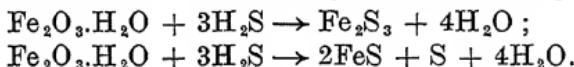
When the foul oxide is removed and exposed to the air, revivification takes place :—

$$2Fe_2S_3 + 3O_2 + 2H_2O \rightarrow 2Fe_2O_3.H_2O + 6S ;$$
$$4FeS + 3O_2 + 2H_2O \rightarrow 2Fe_2O_3.H_2O + 4S.$$
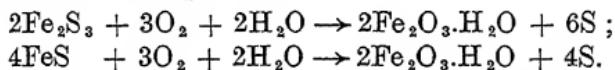
The ordinary type of oxide purifier consists of an open cast iron box having a lute for water cast on the side plates. It is provided with a movable cover made of mild steel, fitted with internal angle and T-iron framing in order to render it rigid. The sides of the cover dip into the water contained in the lute, and so form a seal, which prevents the escape of gas. The cover is provided with suitable fastenings in order to hold it down and prevent the pressure of gas underneath from lifting it up. The purifier is fitted with T-iron bearing bars, on which rest the wooden grids upon which the purifying material is placed. The gas usually enters at the bottom and leaves at the top of the vessel. In order to perform their work efficiently, it is necessary that purifiers should be worked in series. A common arrangement is a series of four, connected by cast

iron pipes and worked by a valve in the centre, enabling any one of the four to be shut off during the operation of emptying the purifier of fouled material and refilling with fresh ; this is not universal. A series of six or more may be employed ; the first four may be worked in rotation, while the last two may be used as " check " vessels to make certain that no trace of impurity slips by.

Another type of purifier, instead of using a water lute, has covering plates made of strong steel and angles, which are bolted to the top of the purifier, being made gas-tight by means of a strip of india-rubber about $\frac{1}{16}$ inch thick. In place of the heavy and expensive lifting gear usually provided, the covers are lifted by a light crane running on rails on the top of the purifier itself, which also forms a floor upon which the men engaged in emptying and charging the vessels can work. When the covers are fastened down the vessel resembles a square or rectangular box. Owing to the absence of a water lute, the purifiers can be worked under greater pressure than those of the ordinary type.

In the most common method of purifying gas, which, as mentioned above, consists of four vessels filled with oxide of iron, the crude gas enters the series of three in use, and when hydrogen sulphide appears halfway in the third vessel, the first one is shut out and a fresh one put in after the third one ; then the purifier originally the second becomes the first one, and thus it is ensured that the third purifier is always a clean one. The oxide purifiers are occasionally followed by two boxes containing lime for taking out carbonic acid. The lime in this case will be found in an offensive condition as carbonate. Lime removes sulphuretted hydrogen, carbon dioxide and carbon disulphide, while oxide of iron removes only the first of these impurities.

Alwyne Meade in *Gasworks Practice* (2nd Edn., London, 1921, 597) gives the following rule for determining the requisite capacity of oxide purifiers :—

For a set of four purifiers allow a superficial area for each

box amounting to 0·5 sq. ft. per thousand cubic feet of gas made per day. If lime purification is also to be resorted to, this allowance should be increased to 0·6 sq. ft., or 0·65 to 0·70 sq. ft. if no catch-boxes are used.

It is said that one ton of fresh, artificial oxide of iron used alone for the elimination of hydrogen sulphide will purify the gas from 200 to 250 tons of coal.

Composition of Gas

The crude gas as it issues from the oven consists of a mixture of permanent gases, while carried in suspension are a number of hydrocarbon and other vapours of varying density, among which is a quantity of water vapour. The chief impurities in the crude gas may be enumerated as follows :—

(1) Suspended tarry matter and condensable hydro-carbons.
(2) Ammonia.
(3) Sulphuretted hydrogen.
(4) Carbon disulphide, and other sulphur compounds.
(5) Carbon dioxide.
(6) Hydrocyanic acid.
(7) Naphthalene.

After the tar and ammonia have been removed and the gas has been cooled to, say, 25° C., an average analysis of the gas may be :—

Hydrogen	56·0–58·0	per cent
Methane	25·5–29·1	,, ,,
Other hydrocarbons	2·5– 2·8	,, ,,
Carbon monoxide	5·0– 5·6	,, ,,
Carbon dioxide	1·9– 2·0	,, ,,
Oxygen	0·5– 0·6	,, ,,
Nitrogen	3·0– 5·0	,, ,,
Inerts	5·0– 7·0	,, ,,
Calorific value	520	B.T.U.
Specific gravity	0·359–0·370	

At the outlet of the primary cooler the gas, in addition to small quantities of suspended tarry matter, contains on an average the following impurities :—

Ammonia . . .	200–300 grains per 100 cubic feet	
Sulphuretted hydrogen	600–900	
Carbon dioxide . .	900–1400	
Other sulphur compounds	35–46	
Hydrocyanic acid .	60–80	
Naphthalene . .	20–25	

A certain percentage of the ammonia is absorbed by the water vapour and is condensed out with the liquor. Water possesses a powerful affinity for ammonia, and at 20° C. and one atmosphere pressure it is capable of absorbing about 740 times its own volume of the gas. Thus, in the indirect method of ammonia recovery, water is used in either scrubbers or washers as a ready means of removing the ammonia.

Measuring, Recording and Governing Instruments

An important factor in heat economy, and an aid in working a coke-oven and by-products plant at the highest point of efficiency, it is necessary to make the fullest use of measuring, recording and governing instruments. It has been stated by Greenfield and Harrison (*C.O.M.A. Year Book* 1929, 193) that a battery should be equipped with at least the following instruments :—

  (1) Total gas volume meter and recorder.
  (2) Surplus gas volume meter and recorder.
  (3) Fuel gas volume meter and recorder.
  (4) Fuel gas pressure recorder.
  (5) Fuel gas pressure governor.
  (6) Stack draught recorder.
  (7) Stack temperature recorder.
  (8) Stack draught governor.

(9) Green gas pressure recorder.

(10) Green gas pressure governor.

(11) Recording gas calorimeter.

(12) Optical pyrometer.

(13) Control clock for automatic gas reversal.

(14) Engine room clocks in the ram cab and coke loco-
motive cab.

(6a) Air pressure recorder, where air is supplied by fan.

(6b) Air volume meter and recorder, where air is supplied
by fan.

(8a) Air pressure governor and recorder, where air is
supplied by fan.

Governors and Gas Regulators (Askania Type).—
On a modern coking plant it is essential that some form
of regulator shall be fixed to the entrance of the main
leading from the ovens, to ensure that the gas is always
drawn from the ovens at a steady and uniform pressure. A
pressure of 2 or 3 mm. of water prevents leakage into the
ovens of the air or flue-gases, which would reduce the calorific
value of the gas. Thus a steady pressure gives the most
economical working and uniformity in the finished products.

Whenever an oven is charged there will be a rush of gas
into the main, and the regulator, being connected to the
exhauster, can increase the speed to draw off this extra
gas and keep the pressure uniform.

A good regulator must be sensitive to small fluctuations
of pressure and react quickly to any sudden change in
conditions ; it must be reliable and as far as possible fool-
proof.

There are a number of good regulators on the market.
The Askania type has a leather diaphragm impulse system
(Fig. 10). Its characteristic component is the hydraulic jet
H, which is arranged to swing horizontally about a vertical
pivot through which the oil flows under considerable pres-
sure. The free end of the jet moves across two adjacent

orifices in the distributor facing the end of the jet. The movement of the jet is obtained from the diaphragm with an increase in pressure, and in the reverse direction from the compressing spring I with a reduction of pressure. This spring, which is adjustable, also serves for setting the regulator to any desired pressure. At the standard pressure for which the instrument is adjusted, the jet of oil from the
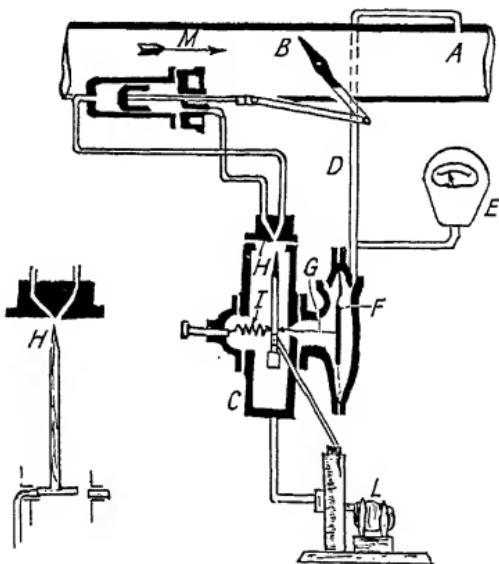
FIG. 10.—Askania Jet Pipe Gas Regulator.

nozzle impinges equally upon the two orifices in the distributor. The kinetic energy of the jet is converted into pressure, and this is equal at each orifice ; there is also equal pressure on each side of the piston in the control cylinder to which the orifices are connected by suitable conduits, which at intervals may require steaming out.

With any deviation from the standard pressure, the jet moves more directly over one or other orifice, and consequently the pressure in the one becomes greater than that

in the other. This difference in pressure is also obtained in the control cylinder, causing the piston to move and adjust the regulating valve to which it is connected. As the pressure again reaches the standard value the jet takes up its neutral position.

This regulator is neat and compact and has a minimum of moving parts, while its principle of action should make it smooth in operation. The power consumption of the oil pump motor is approximately 0·75 H.P.

Electroflo Meters.—The orifice method of flow measurement is one in which cheapness and ease of installation are the main advantages. The rate of flow through an orifice plate is a function of the differential pressure created by the contraction of the fluid stream. In practice two different arrangements of pressure tappings are found. In one, the differential manometer is connected to tappings adjacent to both faces of the orifice plate, and in the other the upstream tapping is about one pipe-diameter up-stream from the orifice plate, the low pressure tapping being about half a diameter down-stream. This arrangement is convenient, but a carrier ring orifice is said to be the more reliable measuring device as it is more standardised. (A fully descriptive paper was given by Dr. Engel, *Gas World*, **107**, 1937, 13–18). The position of the orifice plate should be carefully chosen and there should be a straight length of pipe before and after it. The meter body should be located on a solid foundation and connected to the valves at the main by two $\frac{3}{8}$-inch pipes.

The reading and recording instruments are usually mounted on a panel inside some convenient building. The indicator is an inductance type conductance meter, carrying a maximum of one ampère at full scale reading with a secondary voltage of 36 to 44. There are two coils in the instrument movement, one known as the current coil, which is in series with the other instruments and the meter body,

and the other as the potential coil, which controls the movement of the pointer and makes its position independent of changes of voltage. If the current is entirely switched off, the pointer will swing back past the zero mark; if the current is switched on but the meter body circuit is broken, the pointer will register zero.

The Electroflo Electric Ring Balance Meter.—This meter is a simple means of measuring the flow of low pressure gas with a frictionless positive system of transmitting the readings electrically to the most useful point. The Ring Balance Flow Meter consists essentially of a balanced ring supported at its centre of gravity on knife-edges. A partition is fitted in the upper part of the ring, so that the lower part is partially filled with liquid, and two pressure-measuring chambers result. These are connected by flexible tubes to the incoming differential pressure pipes in the meter housing. When there is a flow in the pipe, the ring is deflected about the knife-edges to a degree which is an exact measure of the differential pressure proportional to the flow rate. As the ring moves, the continuous resistance element supported from it is raised and lowered in the mercury well in the meter housing. The amount of immersion controls the value of the resistance in the electric reading instrument circuit; it therefore increases and decreases the current through the circuit and maintains it always directly proportional to the rate of flow which occurs. The translation of the square root law governing the flow of fluids into the linear law of the easily read electrical indicator, recorder and integrator is accomplished directly, without cams or other motion changing devices, by the fixed dimensions of the resistance element, the ends of the conductors following a suitable contour.

It is of advantage to fix the orifice plate in the seat of the valve so that it can be lifted out for cleaning, as dirty gas can affect the accuracy of this method and cleaning at intervals is necessary.

The Holmes-Connersville Meter.—The Connersville is a displacement type of gas station meter. It consists of two cast iron impellers enclosed within a cast iron casing and supported by means of suitable ball or roller bearings (Fig. 11). The gas enters the top of the meter through the inlet box and exerts a pressure on the impellers which causes them to revolve. For one complete turn each impeller must
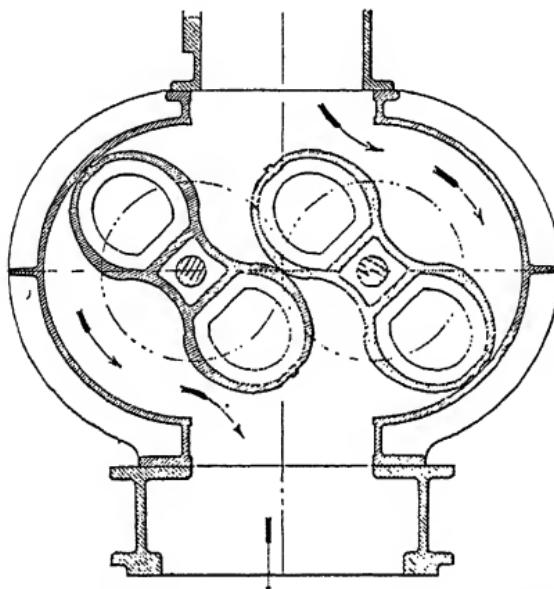
FIG. 11.—Section through a Holmes-Connersville Meter.

pass through its vertical position twice, forming a pocket between itself and the adjacent cylinder each time. Since there are two impellers, four pockets of gas are trapped and released during each revolution of the meter.

Because the impellers are formed of true mathematical curves that follow a well established equation, the displacement of a Connersville meter can be accurately calculated. The cross-sectional area of the space is obtained by subtracting one half of the area of the impeller from one half

of the area of the circle of the cylinder, since the centre lines of the circle and the impeller are coincident. This area multiplied by the length of the meter impeller gives the volume of the space, which multiplied by four gives the displacement for each complete revolution of the meter. The counter is adjusted to convert revolutions of the meter to cubic feet and is coupled direct to the impeller shaft.

One of the largest positive displacement Connersville meters built has been supplied by Messrs. Holmes to the Broken Hill Proprietary Company, Australia. This will measure 31 million cubic feet of coke-oven gas daily. Its normal operating speed is 160 r.p.m. and the displacement per revolution is 138·85 cubic feet.

The Calorific Value of Coal-Gas

The calorific value of coal-gas must be kept at a certain figure if the gas is to be sold to a public gas company. This value is best determined by a recording calorimeter, but it is necessary to check this instrument frequently by spot tests. The instrument usually used and prescribed by the Gas Examiners is the Boys' non-recording calorimeter. Having set up the apparatus, which consists of governor, meter and calorimeter, the meter must be tested by the one-twelfth cubic foot bottle provided, and it must be ascertained that the whole apparatus is gas-tight. This is done by turning on the gas supply tap and clipping the gas tube just before the calorimeter; the meter must not continue to move when tested in this way over a considerable period of time. Water is allowed to pass through the calorimeter, and the gas is then lighted. The speed of passage of gas is adjusted by the governor and supply tap on the inlet side of the meter to give a temperature rise of the water during its passage through the calorimeter of about 20° C. The speed of the water is to be such that when a regular flow of water through the calorimeter has

been established a little may pass the overflow of the funnel and trickle over into the sink. The gas is allowed to continue burning and the water to continue flowing in this way for not less than 45 minutes, at the expiration of which time it is considered that standard conditions will have been established. The operator is then ready to commence the test. When the meter hand is at 75, the inlet temperature is to be read. At the moment the meter hand reaches 100, the funnel is removed so as to direct the overflowing water into the measuring vessel, and at the same time the stopwatch is started. When the meter hand reaches 25 the first reading of the temperature is made, and this temperature is then read every quarter-turn until fifteen readings have been taken. Thermometer readings should be estimated to the nearest one-hundredth of a degree. At every turn of the meter, except the last, a reading should be taken of the inlet temperature during the period when the meter hand is between 75 and 100. At the moment when the meter hand reaches 100, after the last outlet temperature has been read, the funnel is removed so as to direct the outlet water into the sink again, and at the same time the watch is stopped. The meter has then made four revolutions (equal to one-third of a cubic foot) in the time recorded by the stop-watch. Readings are then taken of the barometer and of the thermometers showing the temperatures of the effluent gas, the surrounding air of the room, and the gas in the meter.

The water collected in the measuring vessel is thoroughly stirred and measured, its temperature being taken at the same time. This volume of water must be subsequently corrected for deviation from a temperature of 15° C. Any corrections which are necessary for the thermometers as shown by the certificates must also be made, and the mean of the four readings of the inlet temperature is then subtracted from the mean of the fifteen readings of the outlet temperature. This figure is then multiplied by 11·976 (this

factor includes a correction for the exposed mercury columns
and for the quantity of gas taken—one-third of a cubic foot
—and conversion to B.T.U.). The figure so obtained is
multiplied by the factor for correction of the volume of gas
for temperature and pressure, and by the number of kilo-
grams of water collected at 15° C.

Thus, with the following data :—

Rise in temperature of water . . . . . 21·30° C.
Gas measured at . . . . . 61° F. and 29·9 ins.
  (Gas volume factor is 1·006 from the Gas Referees'
    table)
Weight of water collected (corrected) . . . 1·958 kg.
Temperature of effluent gas . . . . . 66·0° F.
Temperature of air . . . . ·. . . 61·0° F.

we have :

Uncorrected calorimeter result = 1·006 × 1·958 × 11·976 × 21·3
                          = 502·5 B.T.U.

To this figure a further correction must be made on account
of the loss of heat in the effluent gas, since the effluent was
not cooled to the temperature of the air. This correction
is given by the expression :

Difference in ° F. between temperatures of
                          effluent gas and air × 0·4.

In the above case the correction is (66–61) × 0·4 = 2 ;
since the effluent was at a higher temperature than the air,
this correction must be added to the calorimeter result,
making the calorific value of the gas to be 504·5 B.T.U.
gross per cubic foot.

EXTRACTION AND DISTILLATION OF TAR

TAR extraction may be considered under three heads :—

(1) *Condensation*.—When the temperature of the gases is reduced, tar is deposited ; when the gas has been cooled as far as 35° C. approximately, the greater part of the tar has been eliminated.·

(2) *Liquor or Tar Spray*.—This collects the fine globules of tar from the gas, and causes them to fall with the liquor to the bottom, whence they are run to the tar well.

(3) *Tar Extractors*.—Some form of tar extractor is necessary to extract the last traces of tar from the gas, especially in direct ammonia processes, where the gases go direct through the saturators. These may be divided further into mechanical types, such as the Pelouze and Audouin type and the Mallet Tar Extractor, and the electric detarrers.

Mechanical Tar Extractors.—The Pelouze and Audouin type consists of an outer cylindrical cast iron chamber, having a gas inlet and outlet pipe, and an outlet for the condensed liquors. Inside the cylinder is another cylinder of perforated sheet iron, which constitutes the tar extractor or condenser. The sides of this chamber are two thin sheets of perforated iron having a concentric space between, the inner sheet being perforated with rows of holes $\frac{1}{20}$ inch in diameter, and the outer sheet with a series of slots sized 1 by $\frac{1}{8}$ inch. The outer sheet is so arranged as to offer a blank surface opposite the small holes in the inner sheet. The gas and condensable vapours pass through the per-

forations, with the result that the vapours are what may be termed wire-drawn, and striking against the blank space opposite are deposited thereon; from this they flow to the receptacle below and thence to the tar well, while the gas passes on through the slots into the outer cylinder and so to the outlet pipe. The condensing cylinder, as well as circulating slowly, is so balanced as to be capable of acting as its own regulator. This is effected by causing it to move in a hydraulic seal, so that as the make of gas increases or decreases the cylinder rises or falls, with the result that a larger or smaller number of openings are uncovered for the passage of the gas.

Electrostatic Precipitation of Tar.—Essentially, electrical precipitation is a means of applying to the tar fog particles such a force as will expel them from the gas with which they are associated. J. Bradwell (*C.O.M.A. Year Book* 1935, 183) describes an installation designed to treat 8 million cubic feet of gas per day, at a temperature of 30° C., and containing 200 grains of tar fog per 100 cubic feet. An efficiency of 99 per cent. precipitation is guaranteed (fog contents being assessed on a water-free basis), with a power consumption of 4 kilowatts. The precipitation vessel, situated at the outlet of the exhausters, consists of a mild steel shell in which are grouped 85 mild steel tubes, the centre one being $7\frac{3}{4}$ ins. inside diameter, the others 6 ins. inside diameter, and all 9 ft. long. The tubes are expanded at their upper ends into a tube plate which separates the crude inlet gas from the clean outlet gas. A discharge electrode wire, S.W.G. 16, is suspended down the axis of all but the central tube, from a framework which in turn is suspended from a central lead-in rod. Cast iron weights attached to the lower ends of the wires keep them taut, and also support below the tubes a light grid framework having a central socket. Above the shell is a cylindrical chamber divided into an upper and lower section, the latter housing

the lead-in insulator and communicating with the clean gas space. Quick-opening hand holes in the side of the chamber give easy access to the insulator for cleaning and inspection. The lead-in insulator, which is of massive construction, protrudes into the upper section and, as it is wider at the top, is suspended from the base of that section. The lead-in rod, or electrode, which is the subject of patent application, is of 2-inch diameter heavy tubing and extends from the upper chamber to the socket in the framework below the tubes, passing through the lead-in insulator and enlarged central tube. Discharge fins are fitted to the part of the electrode actually in the central tube in order to ensure that the gas passing up this tube is sufficiently cleaned. The central electrode is fixed at two points, *i.e.* in the insulator and in three insulated centreing screws attached to the sides of the top chamber. The top chamber is filled with insulating oil, which perfectly protects the top of the main insulator and the entering insulators from condensation and dust. High-tension current is conveyed from the near-by generators by an ordinary standard high-tension cable, which is led into the top section and connected to the lead-in rod. A wiped joint makes the cable gas- and moisture-tight.

The W.W-D system is a departure from all others and has the advantage that only one insulator is used, with a single suspension rod running through it, and also acting as a lead-in conductor. A feature of electric detarring which has given rise to many breakdowns in the past is the location and type of lead-in insulator used and the means provided for its protection. The standard insulator used in the W.W-D plant is of plain construction without sheddings, and is of special material which is mechanically robust and, unlike porcelain or steatite, has such thermal properties that even spark-over or sudden heating and cooling cannot affect it. A steam coil, automatically controlled, maintains the temperature of the insulating chamber

above the dew-point of the gas, the temperature being indicated at a convenient point by means of a distance thermometer. The narrow neck connecting the insulator chamber to the clean gas chamber is covered by a baffle bell, and by this means the passage of gas currents towards the insulator is minimised.

In general, the more pertinent advantages of the W.W-D precipitator may be summarised briefly as follows :—(1) The lead-in insulator is of special material which is unaffected by even the temperature changes of arc-over. (2) There is only one main insulator and, therefore, one point only at which precautions must be taken. (3) The arrangement of the lead-in suspension and the level of the supporting lead-in insulator allow of all high-tension parts being independent of the external casing, and complete elimination of all fire risks is therefore afforded, even in the unlikely event of insulator failure. (4) The rigid central electrode fixes both the top and bottom framework and entirely inhibits any tendency of the electrode system to swing. In many plants frequent trouble has been experienced from the swinging of the electrode wires, which is the result of relayed impulses, sometimes from electrical conditions and sometimes from mechanical vibrations. (5) The electrode wires are of special alloy steel, highly resistant to surface corrosion and entirely immune from intercrystalline corrosion. Soft iron wires are particularly susceptible to the latter defect, internal stresses being set up and premature breakage resulting.

The equipment includes a motor-driven two-generator set with starter and speed regulators. Being exceedingly compact, it is conveniently housed in a small building beneath the primary gas condensers, which are adjacent to the treating vessel. The current obtained from the generators, being steady, allows an ordinary standard high-tension cable to be used to convey the current to the treating vessel. This cable can be of any length, however, and the treater

need not be near the generating plant. The signal light and warning Klaxon horn are situated in the near-by exhauster house, and the plant is thus under the control of the exhauster attendant.

W.W-D detarrers have varied applications—they have been used on both the pressure side and the suction side of the exhausters and on the inlet to the primary condensers or coolers. The gas has been treated at temperatures ranging from atmospheric to 90° C., while the gas pressure has varied from a few inches suction to a pressure of 70 inches of water. A typical W.W-D detarrer operating on coke-oven gas at outlet exhausters has given the following test result :—Grains of tar per 100 cubic feet of gas (60° F. and 30 ins. of mercury)—inlet average 166, outlet average 0·94, showing an efficiency of 99·4 per cent. The operating potential was 25,000 volts.

Tar Distillation.—Coal-tar is a black viscous fluid of a specific gravity varying from about 1·1 to 1·2. It consists of a complex mixture of compounds, chiefly hydrocarbons, in some cases associated with oxygen, nitrogen, sulphur, etc. The nature of these compounds is largely influenced by the temperature of the coke-oven. The yield of tar varies considerably, being mainly dependent on the nature of the coal and the temperature in the oven. The constituents of tar may be grouped as :—

(1) Hydrocarbons—(a) paraffin series ; (b) benzene series.
(2) Bodies containing oxygen—tar acids, etc.
(3) Bodies containing nitrogen—ammonia, pyridine, etc.
(4) Bodies containing sulphur.
(5) Pitch. ·
(6) Free carbon.

Low Temperature Tar (say of about 500° C. oven temperature) is very fluid and of low specific gravity, rich in paraffin but deficient in benzene hydrocarbons ; it is

rich in tar acids, but has a low content of pitch, about 28 per cent.

High Temperature Tar (say of 1100° C. or over) is of high specific gravity, thick, richer in benzene than in paraffin hydrocarbons, and with a high pitch content, about 64 per cent.

By reason of the difference in the boiling-points of the various compounds in tar, they may be readily split up into groups of compounds by fractional distillation, a rough
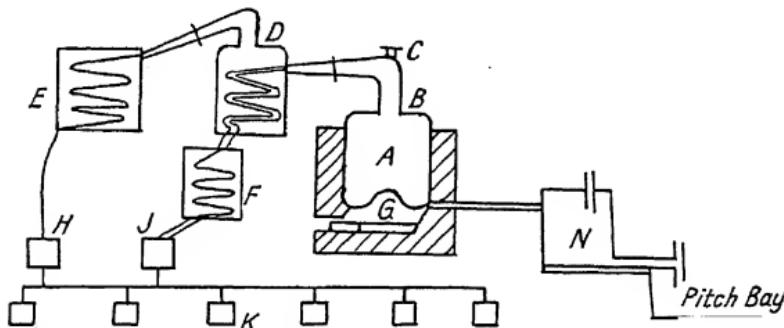
FIG. 12.—Tar Distillation Plant.

separation being obtained by collecting the distillates at the following temperatures to form definite fractions :—

(1) Light oil .   .   collected up to 170° C.
(2) Middle oil   .   „   between 170° and 230° C.
(3) Heavy oil   .   „   „   230° „   270° C.
(4) Anthracene oil   „   „   270° „   360° C.
(5) Pitch   .   .   residue left

A typical tar distillation plant is shown in Fig. 12. The still *A* is constructed of mild steel plates, the bottom being dished to present a greater area to the heat from the grate *G*. The flame after passing under the still rises in a spiral direction round the sides and passes into the chimney flue through an opening fitted with a damper. The vapours
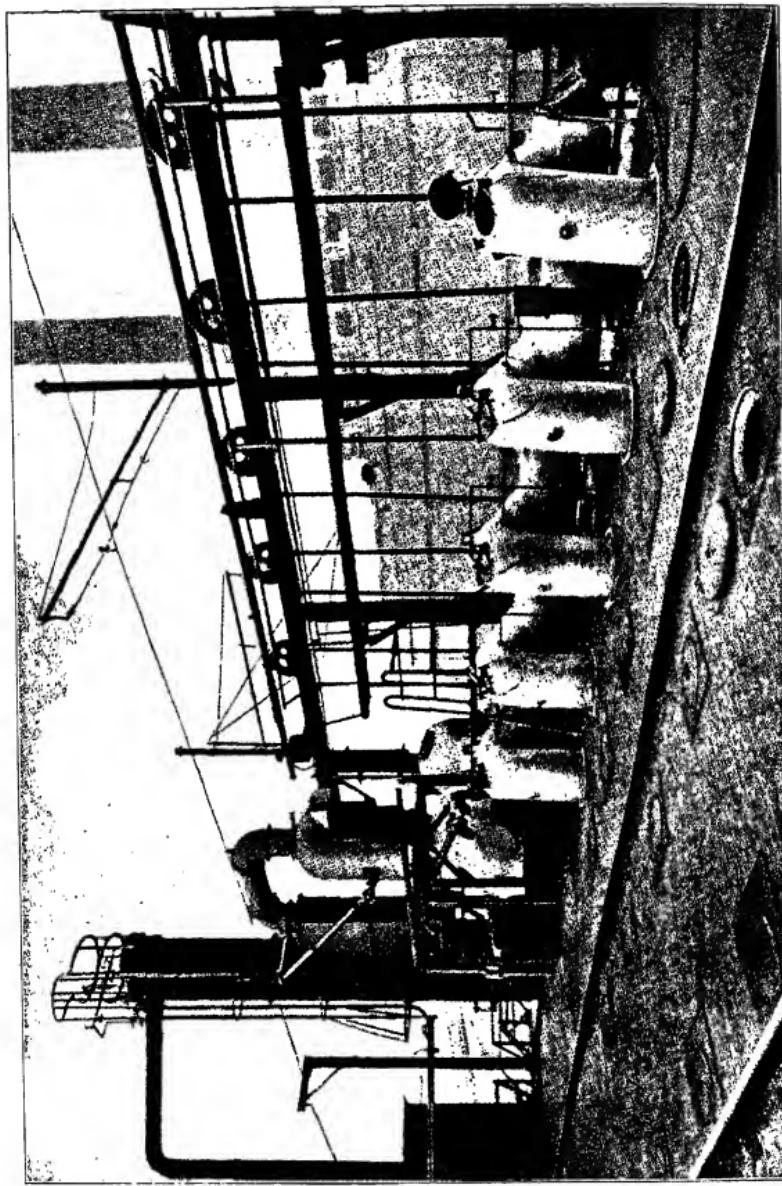
Fig. 13.—Barrett Tar Distillation Process.

evolved pass off through the swan neck B fitted with a
safety valve C, into a coil passing through a primary still
D. This primary still has been previously filled with cold
tar, which is thus preheated by the hot vapours from A.
By thus utilising two stills, considerable economy is effected,
and the water and some of the lighter oils in still D pass
off and are separately condensed in cooler E. The vapours
from the main still A after passing the primary still D are
completely condensed in F. The products from these
coolers E and F pass through inspection overflow boxes H
and J to any desired storage tank K, according to the
fraction desired, the change point being usually determined
by the specific gravity of the distillate. After the light oils
have passed over, the water in condenser F is shut off,
allowing the temperature to rise, since otherwise naphtha-
lene would block the pipes. Successive fractions are taken,
and near the end steam is blown out of the still to expel
the oils of high boiling-point and to prevent the formation
of a residue of carbon in the still. After a few hours the
pitch is run off through N, where fumes pass through a
purifier, and then to the pitch bay when cooled. The main
still is then refilled with hot tar from the primary still D,
which is filled with cold tar, and the process is continued.

   The Barrett Process.—The principle of this process (Fig.
13) consists in the distillation of the tar by means of the
sensible heat in the foul gases issuing from the ovens up the
ascension pipes.

   The carbonisation of one ton of coal in the ovens is said
to be sufficient to distil upwards of 100 gallons of tar, and
for the average coke-oven plant the whole of the tar made
can be distilled by the hot gases rising from five or six ovens.
It is further said that the process is so flexible that hard
pitch containing less than 40 per cent. of volatile matter,
pitch of ordinary melting-point, or road tar can be made
of any desired fraction.

CHAPTER VIII

RECOVERY OF AMMONIA

THE ammonia in coke-oven gas may be recovered in the form of sulphate of ammonia, or as aqueous ammonia of approximately 25 per cent. strength.

The principle involved in the manufacture of sulphate of ammonia is the combination of ammonia and sulphuric acid. To get the maximum output of sulphate of ammonia it is essential to keep cool the oven tops, ascension pipes and mains, and to see that the ovens are well filled, for the decomposition temperature of ammonia is 750° to 800° C. It is necessary to withdraw the gas from the ovens with the minimum of contact with hot surfaces which might crack the ammonia.

The following are methods of producing sulphate of ammonia :—

(1) The Indirect Method.
(2) The Semi-direct Method.
(3) The Direct Method.

These are the methods used at coke-oven plants and gasworks.

(4) The Synthetic Method, which hardly comes under Coke-oven By-products, although coke-oven gas may be utilised.

The Indirect Method.—In this method the ammonia is taken from the gas in two ways :—

(a) The liquor which condenses from the gas passing the

cooling plant contains ammonia, chiefly fixed ammonia.

(b) Ammonia liquor is produced by scrubbing the gas with water.

These two are then usually mixed together making a liquor containing approximately 1·0 to 1·8 per cent. of ammonia. On a plant producing, say, 12 tons of sulphate of ammonia daily, approximately 300 tons of liquor have to be treated. This liquor is generally first preheated by passing through preheaters which are heated by steam and gases rising from the saturator. It is then run into one of the top trays of an ammonia still, whence it passes into a lime mixing apparatus where it is intimately mixed with milk of lime, perhaps 10 to 20 per cent. by volume being added to the ammonia liquor, which then passes back to the next lower tray of the still. As the liquor passes down the still, steam bubbles up through it and extracts ammonia from it. The steam takes out the free ammonia, while the lime extracts the fixed ammonia.

By *free ammonia* is meant the ammonia in combination with the two weak acids, carbon dioxide and sulphuretted hydrogen, the compounds so formed being decomposed at a temperature below that of boiling water, so that the ammonia can be expelled from the solution by the simple act of boiling. By *fixed ammonia* is meant the ammonia in combination with certain other acids, the salts so formed not being decomposable by simple boiling, but requiring to be heated with an alkaline substance, such as lime, for which acids have a strong affinity.

The still comprises a series of superimposed sections, each having removable doors for cleaning purposes, and each having a steam distributing passage over which is placed a toothed hood. Each section has an overflow weir and a sealing plate, each of which alternate in such a manner that the liquid descends in zigzag fashion, passing horizontally

across each tray in turn, while the steam and vapour rise through the liquor in the passages and hoods of the sections to the top of the still. In this case, the liquor, on reaching the fourth tray from the top of the still, passes to the bottom of the external liming chamber. Here it is mixed with milk of lime, the two liquids being agitated and brought into intimate contact by means of steam, and re-enters the still at the fifth section ; it passes downwards in a zigzag fashion, meeting an ascending current of steam. The vapours of ammonia and steam rise to the top of the still and thence travel to the saturator, where they pass through an acid bath and sulphate of ammonia crystals are formed.

The ammonia vapours thus rise to the top of the still and pass to the saturator mixed with steam at a combined temperature of about 100° C. In the saturator is a boiling sulphuric acid solution at a twaddell of from 56° to 63°. The twaddell must not be allowed to go below 56° or blue salt may be the result. Usually a twaddell of about 57° is aimed at, as a mean, to make a good salt ; a continuous stream of acid is run into the saturator to keep the bath at the twaddell decided by the management. Crystals of ammonium sulphate form in the bottom of the bath of the saturator, and are ejected by steam or compressed air on to a draining table, where the sulphate is retained and whence the surplus liquor drains back to the saturator. The sulphate is then transferred to the basket of a centrifugal drier (Fig. 14), where it is whizzed for some minutes, and then in a fairly dry state the acid salt is dropped into some type of neutraliser and drier, for sulphate of ammonia must be produced in a neutral and dry state, and in as good a grain as possible, to meet the requirements of the market.

Every few days, as required, the saturator bath is made up to over 64° Tw. with acid. This has the effect of cleaning the saturator and preventing the formation of rock salt. For general working and production of large crystals, a twaddell of about 57°, or a free acid content of about 4·0

per cent., is usually worked at. If the crystals are deposited when the twaddell is high, say, 65°, they are in too fine a state, and are somewhat slimy and apt to hold the acid and moisture. It is best to adjust the acid flow so as to maintain a specific gravity of 56° to 57° Tw. continuously. As a check, the free acid in the bath should vary between 3·0 per cent. and 6·0 per cent. These conditions should
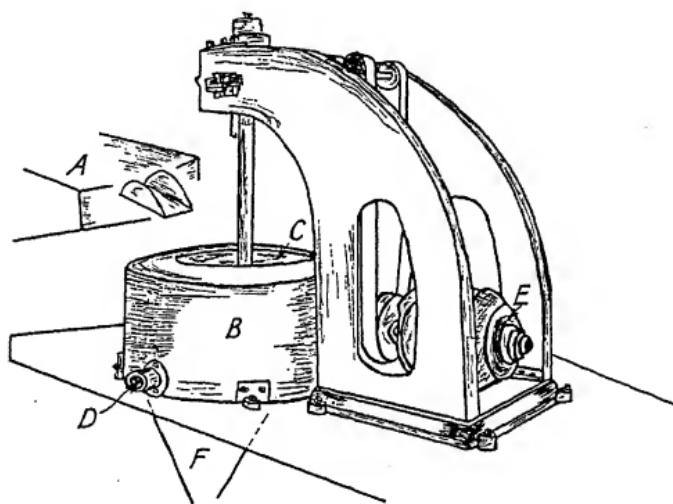
A.	Draining table.	D.	Liquor drain to saturator.
B.	Whizzer body.	E.	Motor.
C.	Revolving basket of perforated copper.	F.	Chute for sulphate to neutraliser.

Fig. 14.—Centrifugal Drier for Sulphate of Ammonia.

result in a white sulphate with large crystals. Blue sulphate may be produced if the specific gravity of the saturator drops below a twaddell of 54°. This causes the iron salts in the acid to precipitate and combine with cyanogen compounds, and become fixed as ferrocyanides. On the admission of further acid, the ferrocyanide is converted into ferrous ferrocyanide by the sulphate of iron. This ferrous salt on exposure to the atmosphere is oxidised to Turnbull's

blue or Prussian blue. This may not be apparent at first, but may develop in a few days.

The indirect method of making sulphate of ammonia requires careful supervision, both night and day, or great losses may occur. The following tests should be taken at regular intervals, at least once per shift :—

(a) Test for ammonia in the gas after scrubbing. If more than 2·5 grams of ammonia per 100 cubic metres of gas are found, then more water is needed on the scrubber, a lower temperature is needed, the scrubber itself requires cleaning, or some detail of operation needs attention.

(b) Temperatures of gas and water before and after scrubbers.

(c) Strength of ammonia liquor made.

(d) Waste liquor from the bottom of the still should not show more than 0·002 per cent. of ammonia or the still is not working correctly. If fixed ammonia is present then more lime is required. If free ammonia, then more steam is necessary.

(e) Devil liquors and gases for traces of ammonia.

(f) Temperature of ammonia gas entering the saturator.

(g) Twaddell and acidity of saturator bath.

(h) Acidity or otherwise of sulphate of ammonia made.

The *advantages* of the indirect method of sulphate recovery are :—

(1) General elasticity of the process ; stoppage for repairs easy if liquor storage available.

(2) Easy conversion into processes for the manufacture of alternative compounds.

(3) Smallest exhauster and least power for tar extraction required of the three systems.

(4) No naphthalene extraction plant required.

The *disadvantages* are :—

(1) Liability of ammonia loss :—(a) at scrubbers, (b) by

evaporation from storage tanks, (c) at still in waste
liquors.

(2) High labour, working and repairs costs.

(3) High consumption of steam, approximately 8–10 tons
of steam per ton of sulphate of ammonia made.

(4) Waste liquors produced often difficult to dispose of ;
largest effluent of the three methods.

(5) Liability of making discoloured sulphate through
(a) faulty working of still or (b) irregular working
of saturator.

(6) Needs ammonia scrubbers and the largest ammonia
still of the three systems.

The Semi-direct Method of ammonia recovery was
commenced in order to obviate the excessive quantity of
waste liquors obtained by the indirect method. The first
difficulty is to remove the last particles of the tar before
the gas enters the saturator, or dark-coloured salt is the
result. Therefore, after the primary coolers the gas must
be passed through some type of efficient tar extractor, before
being passed at a temperature of about 35° C. direct through
sulphuric acid in a large, enclosed saturator, thus obviating
scrubbers and the necessity for a large amount of water.
But as there is always a certain amount of water in coal,
there is always a certain amount of ammoniacal liquor
produced with the tar which requires distillation at intervals
in a smaller still, similar to that used in the indirect process.
The ammonia gas produced is piped into the main flow of
gas just before the saturator. Thus half the ammonia is
recovered direct from the gas, and the other half by the
ordinary distillation method—hence the name "semi-
direct." In this process, there is a greatly reduced quantity
of effluent, due to the lesser amount of liquor to be distilled,
and also a great saving of steam. Scrubbing is dispensed
with—another saving of steam, water and plant. Other-
wise the same attention is required, with the addition of

passing the whole of the gas through the saturator bath.
The *advantages* of the semi-direct method are :—

(1) Reduced capital cost, by elimination of scrubbers,
    pumps, etc.
(2) Saving in steam and water.
(3) Reduced effluent for disposal.

While the *disadvantages* may be :—

(1) Personal attention still important.
(2) An effluent still remains.
(3) Fairly large still needed.
(4) Large saturator required.
(5) The gas has to be cooled twice, first for tar extraction
    and again prior to benzole scrubbing.

The Direct Method.—In this process sulphate of am-
monia is produced without the production of ammoniacal
liquor. The hot gases pass direct through the saturator
(Fig. 15). The important points of the direct process are
as follows :—

(1) The satisfactory elimination of tar from the hot crude
    gas without the d position of ammoniacal liquor.
(2) The elimination of ammonium chloride where it is
    present to any extent.
(3) The removal of naphthalene and light oils from the
    gas after the gas has left the saturator.

To remove tar satisfactorily from hot coal gas without
the condensation of water, it is obvious that the gas must
not be cooled down below its dew-point, which varies accord-
ing to the degree of saturation of the gas with water vapour
—it is generally below 80° C. and gas may be cooled to
80° C. for the tar extraction process. At this temperature
the bulk of heavy tar will condense out of the gas and can
be removed by simply passing the gas through a static
apparatus of such a design as to cause the small particles

of tar to collect together and combine to form a stream or film of tar, which can be drained away. An electrostatic detarrer is now often used.

Owing to the temperature at which the tar extraction takes place, the gas entering the saturator contains a

A. Gas inlet.	D. Seal.
B. Gas bubbler.	E. Sulphate ejector.
C. Bath of liquor.	F. Gas outlet with Baffles.
	Perforated steam coil.
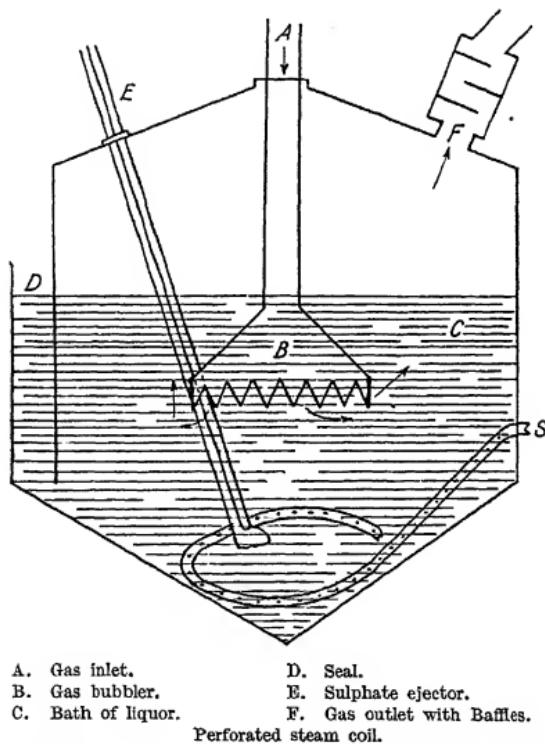
Fig. 15.—Sulphate of Ammonia Saturator, Lead Lined Enclosed Type for Direct and Semi-direct Plants.

quantity of light oils and naphthalene, which, however, pass through the saturator practically unaltered and are removed without difficulty in the secondary coolers before the benzole scrubbers. In the Simon-Carves direct process this separation is attained by washing the gas when at about 40° C. with a limited amount of creosote oil. In the Otto direct

process the naphthalene is removed by passing the gas upwards through a tall scrubber without grids, where it is washed by a falling spray of cold water ; the naphthalene is separated from the gas by the sudden shock of cooling.

Thus in the direct process the gas is only cooled to 80° or 85° C., and the hot gas containing as much free ammonia as possible passes to the saturator after tar extraction.

The *advantages* of direct recovery are as follows :—

(1) The great saving in ground space and apparatus—pumps, scrubbers, stills, storage tanks, etc.
(2) No ammoniacal liquor for distillation.
(3) Large saving of steam due to the absence of pumps and still.
(4) Absence of waste liquors of disagreeable character.
(5) Increased yield of sulphate due to the elimination of evaporation losses.
(6) Reduced cost in upkeep and saving of labour, due to compactness and simplicity of plant.
(7) The direct process makes it possible to separate the ammonium chloride from the gas, and subsequently recover such chloride by simple evaporation, thus producing a valuable salt at a low cost.

The *disadvantages* of the direct process are :—

(1) Largest exhauster of the three processes.
(2) Naphthalene extracting plant needed.
(3) Large saturator required.

The British Sulphate of Ammonia Federation require a neutral sulphate of ammonia as follows :—

(1) Free running grain and not lumpy.
(2) Neutral—must contain under 0·025 per cent. free acid.
(3) Moisture content to be under 0·20 per cent.
(4) Nitrogen to be at least 20·60 per cent.

The British Sulphate of Ammonia Federation control and organise the sale of sulphate of ammonia, and extra is paid

for additional nitrogen content ; deductions are made for acidity and for poor nitrogen content.

Large-Grain Crystals of Sulphate of Ammonia.— There now appears to be a growing demand for large-grained sulphate (or rather for a sulphate with less dust, below 72 B.S.S. size). It appears probable that in future a higher price will be paid for a Grade 1 sulphate of ammonia, which must contain less than 5·0 per cent. dust below 72 B.S.S. Many coking plants are experimenting to eliminate as much dust as possible, by allowing the sulphate to remain longer in the saturator so that the crystals may grow larger, or by fixing some stirring or circulating device to move the grains or crystals about in contact with the liquor. The addition of oxalic acid has also been tried in small quantities. The British Sulphate of Ammonia Federation are endeavouring to standardise both the crystal size and the colour. This, it is hoped, will increase our export sales, by improving and standardising the quality of British neutral sulphate of ammonia.

Liquid Ammonia from Coke-oven Liquids.—Instead of making sulphate of ammonia, the management may consider it advisable to make liquid ammonia of a strength of approximately 25 to 28 per cent. ammonia. One such method is first to pump the crude ammoniacal liquor into an overhead storage tank, fitted with liquor overflow and a tar run-off pipe. Care should be taken to ensure that the crude liquor is as free as possible from tar before pumping to the overhead storage tank, otherwise it may cause trouble. It is then passed through a superheater, where its temperature is raised to about 30° C. before passing down the ammonia still, where it is treated with lime and steam ; the last traces of ammonia are extracted from the liquid, which is allowed to pass away at the still bottom through the usual ball float valve to a discharge pipe.

The hot ammonia gases, along with some impurities, rise from the top of the still and pass through the superheater, where some of the heat is given up to the incoming crude liquor. The gases now enter the bottom chamber of the reflux tower (which is similar to the ordinary still) and pass up the reflux condenser, which consists of a tube water cooled condenser. The temperature of the outgoing gases may thus be controlled, and any condensate separating out of the bottom of the tower is pumped back into the still. The gases are now led through an atmospheric condenser made with 2-inch steam piping, up through a soda washer, containing a strong solution of caustic soda to remove all traces of sulphuretted hydrogen, then through the second and third washers, containing oil and potassium permanganate respectively for the complete removal of pyridine. The pure ammonia gas is then absorbed in a saturator which consists of a large closed tank partly filled with water, usually three of these saturators being arranged so that any one of them may be used.

CHAPTER IX

RECOVERY AND PURIFICATION OF BENZOLE

In modern practice the recovery of benzole, either crude or as a purified product, is one of the most valuable functions of a coking plant. In oven gas there is a mixture of aromatic compounds, which are extracted by treating the gas with an oil (a solvent of relatively high boiling-point) in scrubbers. These aromatic compounds are hydrocarbons of the general formula C_nH_{2n-6} and are associated with certain impurities.

The *main products* are :—

	Formula.	Boiling-point.
Benzene	C_6H_6	80·5° C.
Toluene . ·. . . .	C_7H_8	110·0° C.
Xylene, *ortho-, meta-, para-*	C_8H_{10}	142°, 139°, 138° C
Propyl-benzene . . .	C_9H_{12}	160° C.

The *impurities* are :—

	Formula.
Carbon disulphide	CS_2
Thiophen	C_4H_4S
Thiotolen	C_5H_6S
Phenols	$C_nH_{2n-7}OH$
Paraffins	C_nH_{2n+2}
Pyridine	C_5H_5N
Naphthalene	$C_{10}H_8$

Creosote oil and other resinous matters.

Crude benzole is recovered from the gas by passing it up a series of tower scrubbers in counterflow to an absorbent

wash oil, which is sprayed on to the top of each scrubber and percolates downwards through a series of wooden grids, similar to those in an ammonia scrubber. The gas thus meets a large surface of grids wetted with the absorbent oil, and is also washed by the oil itself, as it rises up the scrubber. Usually either two or three scrubbers are utilised, and the flow of the oil is so arranged that the fresh oil meets the gas as it goes up the third scrubber, thus scrubbing from the gas the last traces of benzole hydrocarbons.

The efficiency of benzole recovery by oil washing is bound up with absorptive capacity and rates of absorption. J. Curry (*C.O.M.A. Year Book* 1938, 141) describes the use of creosote oil, a mixture of creosote and anthracene oil, anthracene oil and gas oil. He states that anthracene alone resulted in frequent changing of the oil due to high viscosity, and that a mixture of anthracene and blast-furnace creosote also caused a blockage in the scrubber.

ABSORPTIVE EFFICIENCIES OF WASH OILS FOR BENZOLE (CURRY)

Fresh Wash Oil.	Distillation Range (distillate up to 200° C. removed).		Specific Gravity at 15° C.	Viscosity at 70° F., Redwood No. 1.	Absorption of Benzole.	
					Gms. per 100 gms. of oil.	Vols. per 100 vols. of oil.
1. Creosote (redistilled) . . .	86·5 per cent. at 300° C.		1·007	36	3·44	3·75
2. Mixture of creosote and redistilled creosote .	82·0	,, ,, 300° C.	1·045	38·5	3·42	3·72
3. Creosote . .	80·0	,, ,, 300° C.	1·059	40	3·24	3·65
4. Anthracene oil .	83·0	,, ,, 385° C.	1·120	130	2·54	3·15
5. Gas oil . . .	97·0	,, ,, 300° C.	0·850	40	3·50	3·30

He got good results using No. 2 or a mixture of creosote and redistilled creosote to the following specification :—

Specific gravity . . . . . . 1·04–1·050
Drop point . . . . . . 205°– 210° C.
Proportion distilling up to 230° C. . . 15·0–20·0 per cent.
    ,,   ,,   ,, 270° C. . . 60·0–65·0 ,, ,,
    ,,   ,,   ,, 300° C. . . 80·0–85·0 ,, ,,
Viscosity at 70° F., Red. No. 1 . . . 39–40 seconds
   ,,   ,, 70° F. after distilling off front
    ends to 230° C., Red. No. 1 . . . 45 seconds
Naphthalene not to exceed 12·0 per cent. on steam distillation

Using cresote as a washing oil, the trouble that older plants used to experience with oil thickening has been largely overcome as a result of the gas now being freed from tar fog by electric precipitation.

White and Kellet (*C.O.M.A. Year Book* 1933, 215) have given a comparison of the two wash oils in general use, namely, creosote oil and gas oil.

The specifications of the oils used were :—

	Gas Oil.	Creosote Oil.
Specific gravity	0·8625	0·9980
Viscosity at 20° C., Red. No. 1, secs. . .	42·0	40·5
Distillation range (bar. press. 744·1 mm.) .		
Percentage by volume distilling up to 200° C. .	0	0
,, ,, ,, ,, ,, 223° C. .	0	0
,, ,, ,, ,, ,, 225° C. .	0	12·5
,, ,, ,, ,, ,, 250° C. .	14·5	49·5
,, ,, ,, ,, ,, 275° C. .	37·5	67·0
,, . ,, ,, ,, ,, 300° C. .	56·5	78·0
,, ,, ,, ,, ,, 325° C. .	72·0	87·0
,, ,, ,, ,, ,, 350° C. .	83·0	94·5
,, ·,, ,, ,, ,, 375° C. .	91·0	—
Tar acids	—	trace
Naphthalene (by picrate method), per cent. by weight	—	17·8

ABSORPTIVE CAPACITY

At 40° C.	1·3	1·55
,, 30° C.	1·9	2·40
,, 20° C.	3·0	3·8
,, 10° C.	4·8	6·3
·· 0° C.	8·15	10·9

These workers state that the ideal absorbent is one that possesses the following properties :—(1) high absorptive capacity, (2) low viscosity, (3) high initial boiling-point, and (4) low specific heat. It would seem that creosote oil, having a higher absorptive capacity, is more advantageous to use. Gas oil has, however, the following good points :— (1) lower viscosity at low temperatures ; (2) absorptive capacity tends to rise whilst in use ; (3) freedom from naphthalene ; (4) lower consumption of oil per gallon of benzole ; and (5) lower cost.

Temperature has a great deal to do with efficient benzole recovery. The temperature both of the gas going into the scrubbers and of the wash oil before the scrubbers must be carefully controlled. The gas, as far as possible, should be cooled down to at least 20° C. before it enters the scrubbers, and the wash oil should be only one or two degrees higher. Usually three benzole scrubbers are used, sometimes only two ; these are generally from 75 feet to 120 feet high and from 10 to 20 feet in diameter, and are filled, as are ammonia scrubbers, with wood grids. The fresh, debenzolised oil is pumped on to the top of No. 3 Scrubber in the gas stream, whence it is sprayed on to the top of the grids, through which it passes, coming into intimate contact with the partly stripped gas ; it is then pumped on to No. 2, and finally on to No. 1 Scrubber. The gas, on the other hand, rises first up No. 1 Scrubber, then up No. 2 and finally up No 3. It is thus seen that the fresh oil meets the almost stripped gas in Scrubber No. 3, taking the final traces of benzole from it ; this oil, containing a little benzole, then comes down No. 2 Scrubber, meeting partly stripped gas ; and it finally comes down No. 1 Scrubber, where it meets the fresh, strongly benzolised gas flow.

Daily tests should be taken of the amount of benzole remaining in the gas after passing the scrubbers. If too much, then probably a greater flow of oil is needed, or the

temperature of either the gas before the scrubbers or the wash oil requires lowering.

At intervals these scrubbers may be blocked, the boards may have collapsed, or become clogged with sludge, and new boards may have to be put inside the scrubber ; or the scrubber may require steaming to remove the blockage. With such blockages, the scrubber would not be working correctly, and this would show both in back pressure tests and in the daily after-scrubber test of the gas.   S. Tweedy (*C.O.M.A. Year Book* 1938, 149) describes a method of removing scrubber blockages by circulating about 1000 gallons of crude benzole around the scrubber for 8 to 12 hours.

Too low a temperature of the wash oil has been said to result in a reduced recovery of benzole, and too warm a wash oil has a tendency to give up naphthalene to the gas.   If the oil is much cooler than the gas, condensation of water vapour will be caused in the scrubber, with consequent difficulties in working.   The best results are said to be achieved with the temperature of the gas at about 19° to 20° C. before the scrubbers ; and the temperature of the wash oil reduced at the oil coolers after the crude still to 21° to 22° C.

With efficient scrubbers a good recovery of benzole may be obtained by the use of 80 to 120 gallons of wash oil per 12,000 cubic feet of gas, or per ton of coal carbonised approximately.

The benzolised oil, after passing down the scrubbers, passes to the benzole distillation plant for the extraction of benzole, which is carried out by the application of heat. The oil passes through heat exchangers, where it absorbs heat (1) from hot debenzolised oil and (2) from tubes through which pass hot benzole vapours and steam rising from the crude still.   This may raise its temperature some 50° C. or more before it passes through the preheaters, where it comes into contact with steam coils, which raise its temperature to 120° to 130° C. before it enters the crude benzole still.

In the still it passes over successive trays, as in the ammonia still, and is treated with live steam, which agitates it and expels the benzole fumes. These fumes then rise up through the preheater, where they are partly cooled by contact with the incoming benzolised oil tubes. The vapours then pass to the condenser, where they are cooled, coming into contact with water cooled pipes, and are finally condensed. The mixture of water and crude benzole passes to the separator, in which the water is siphoned off from the bottom, and the crude benzole is run off from the top to a storage tank. The hot debenzolised oil passes through the preheater, where it gives up some of its heat to the incoming benzolised oil and then passes through the coolers (probably annular atmospheric type aided with sprays of water on the tops of the pipes). These coolers should cool the oil to approximately 22° to 25° C. Thence the oil passes to a storage tank for the scrubbing system again.

A test should always be made of the benzolised oil before the still, and of the debenzolised oil afterwards. These tests, in conjunction with the after-scrubber test, will give the manager some idea of the efficiency of his plant.

It is important to see that the still has done its work of thoroughly stripping the oil free from benzole properly, so as to ensure that the benzole is taken from the gas by the scrubbers. The effect of incompletely debenzolising the oil, whereby partly benzolised oil is returned to the scrubbers, is very similar to that of over-saturation during the washing of the gas. Failure to strip the whole of the crude benzole from the oil may be due to the following :—(1) too low a temperature of the oil entering the still, (2) insufficient live steam, (3) low time of contact in the still, and (4) too drastic fractionation.

It is also important to note that there is no water in the wash oil, as this is a cause of sludge in the oil ; further, water may be a sign of leaking tubes in the heaters. All ammonia should be removed prior to the benzole scrubbers,

as if it is not completely removed any water in the wash oil absorbs the ammonia compounds, which are decomposed in the steam preheater, the liberated acids attacking the heating coils, more especially if they are of steel or wrought iron (cast iron coils are the most resistant). Water in the wash oil from a perforated heating coil may give rise to a sudden increase in the amount of steam, with a consequent priming of the still.

To produce commercial products, such as 90 per cent. benzole and solvent naphtha, the crude benzole is submitted to further purification and redistillation.

Benzole Rectification

Crude benzole, as recovered, consists of the lower benzole fraction, a heavier fraction of solvent naphthas, and the wash oil which is unavoidably carried over from the crude wash oil still. A fractional distillation in a pot-still will separate the above three fractions of the crude benzole, the cut points being predetermined by laboratory experiment. The first fraction to distil is called *once-run benzole,* and this is the fraction from which a refined motor spirit is made. The second fraction is the solvent naphtha and the heavy solvent naphtha, taken as one. When this has been distilled over, the wash oil is left as residue in the still ; this is run off into receptacles while hot and allowed to cool for the separation of naphthalene. If necessary, the two naphthas may be separated by a further distillation.

The once-run benzole, giving a distillation figure of between 90 and 95 per cent. at 120° C. (retort), against one of 65 per cent. for the crude benzole, is then washed in a washer before final rectification and distillation. The method of washing varies slightly on different plants, but may be as follows :

Charge to washer—approximately 3000 gallons, weighing about 12 tons.

Acid used—strength approximately 160° Tw., 92–94 per cent. H_2SO_4 ; weight 1,300 lbs.

Soda used—mixture of 125–215 lbs. of soda-ash and 28–45 lbs. of caustic soda (approx. 81 per cent. and 19 per cent.) boiled up with 1000 gallons of water in a tank heated by a steam coil ; final strength of mixture 23° Tw. at 60° C.

The washer is filled and allowed to stand 20 to 30 minutes, and any separated water is run off.   The agitator is started and brought gradually to its highest speed, when 1·04 per cent. (of the total charge) of acid is run in from a convenient tank, and agitation continued for half-an-hour.   The liquid is allowed to settle for about half-an-hour, when the acid tar formed is run off, care being taken that no benzole is lost.   The agitator is again started and a further 1·3 per cent. of acid added.   This is agitated for half-an-hour, allowed to settle for half-an-hour, and the acid tar again run off.   After a further ten minutes' agitation, followed by settling, some more acid tar may be removed.   This latter results from particles of the tar held in suspension by the benzole ; the slightest agitation forces them together to form larger particles, which sink to the bottom of the washer.

Note.—The benzole should be tested by the usual washing tests at this point and if necessary a further quantity of acid may be added.

The agitator is again set in motion and 1·8 per cent. of the soda mixture is run in and agitation continued for from three-quarters to one hour.   After the liquid has settled for three-quarters of an hour, the soda layer is run off.   This may be insufficient to neutralise the acid in the charge, in which case a further quantity of soda is added.   Tests should be carried out at every washing to show whether neutralisation has taken place.   During the soda washing the agitator should not be run briskly, as this causes emulsi-

fication of benzole and water. The liquid should be washed gently with water, the agitating device being kept just moving slowly (if at all). Then a hose with a fine spray may be played over the surface of the charge, and down the sides of the washer. Emulsification should be strongly guarded against. The water should be allowed to settle for at least one hour (two hours if possible) before it is run off. The benzole is now ready for final rectification.

Benzole Rectification using Cresolic Acid.—All the " once-run benzole " is first washed with $2\frac{1}{2}$ per cent. B.O.V. acid, then with a soda wash, and finally with water. (The wash is made up in the same way and used in the same proportions as mentioned at the beginning of this chapter.) The cresolic acid is made up for use by mixing $\frac{1}{2}$ gallon of a 50 per cent. by weight mixture of catechol and methanol with $1\frac{1}{2}$ gallons of cresol (the cresol solution then contains 10 to $12\frac{1}{2}$ per cent. of catechol). One to two pints of this solution, mixed with benzole and made up to 1 gallon, is then dribbled into the distillation of approximately 5000 gallons of once-run, *i.e.* 4200 gallons of motor spirit. The distillations may be worked in a series of five per shift, and to the fifth $\frac{1}{2}$ pint extra is added.

1932 Specification for Motor Benzole

1. *Colour.*—The benzole shall not be darker than a freshly prepared solution of 0·5 ml. of 0·1 N. iodine solution in 1000 ml. of distilled water.
2. *Specific Gravity.*—The specific gravity of the benzole at 15·5° C./15·5° C. shall not be lower than 0·8700, nor higher than 0·8850.
3. *Water.*—The benzole shall be free from water and other visible impurities at 15·5° C.
4. *Distillation.*—When 100 ml. of the benzole are tested by the standard method, the volume of distillate

collected when the temperature has reached 100° C. (running-point) shall not be less than 60 ml. ; and when the temperature has reached 120° C. (running-point) at least 85 ml. shall have distilled. When the temperature has reached 145° C. (stop-point) the volume of distillate shall be at least 95 ml. The residue in the flask at the end of the distillation shall remain liquid when cooled to 0° C. for 30 minutes.

5. *Rectification Test.*—The benzole shall pass either the standard acid washing test or the standard oxidation test.

   (*a*) *Acid washing.*—When the benzole is tested by the standard method, the acid layer which separates shall be clear, shall not be darker than a solution of 5·0 gms. of pure potassium dichromate in 1000 ml. of a mixture of equal volumes of pure sulphuric acid (containing 98 per cent. by weight of H_2SO_4) and distilled water.

   (*b*) *Oxidation test.*—When the benzole is tested by the standard method, the weight of residue which is obtained shall not exceed 8 mg. per 100 ml. of the benzole.

6. *Total Sulphur.*—The benzole shall not contain more than 0·4 per cent. by weight of total sulphur.

7. *Free Sulphur.*—The benzole shall not give a steel-grey or black discoloration when tested by the standard method.

8. *Freedom from Acids, Alkalies and Sulphuretted Hydrogen.* —The benzole shall not give a positive reaction for acids, alkalies or sulphuretted hydrogen.

9. *Odour.*—The benzole shall have a pronounced odour of the aromatic hydrocarbons.

10. *Crystallising-point.*—The crystallising-point shall not be above − 5° C.

1929 Specification for 90's Benzole

Similar to motor benzole except :—

1. *Colour.*—Not darker than a solution of 0·2 ml. of 0·1 N. iodine in 1000 ml. of distilled water.
2. *Specific Gravity.*—0·8775 to 0·8835.
3. *Distillation.*—When 100 ml. are tested by the standard method, 90 to 93 ml. shall have distilled at 100°C. (stop-point), and at least 97 ml. when the temperature has reached 120° C. (stop-point).
4. *Water.*—The benzole shall be free from water and other visible impurities at 15·5° C.
5. *Acid Washing.*—When the benzole is tested by the standard method, the acid layer which separates shall be clear, and shall not be darker than a solution of 1·5 gm. of pure potassium dichromate in 1000 ml. of a mixture of equal volumes of pure sulphuric acid (containing 98 per cent. by weight of H_2SO_4) and distilled water.
6. *Total Sulphur.*—The benzole shall not contain more than 0·4 per cent. by weight of total sulphur.
7. *Free Sulphur.*—The benzole shall not contain free sulphur.
8. *Freedom from Acids, Alkalies and Sulphuretted Hydrogen.*—The benzole shall not give a positive reaction for acids, alkalies or sulphuretted hydrogen.

1929 Specification for Pure Toluole

1. *Colour.*—The toluole shall not be darker than a freshly prepared solution of 0·2 ml. of 0·1 N. iodine solution in 1000 ml. of distilled water.
2. *Specific Gravity.*—The specific gravity of the toluole at 15·5° C./15·5° C. shall not be lower than 0·8665 nor higher than 0·8715.
3. *Water.*—The toluole shall be free from water and other visible impurities at 15·5° C.

4. *Distillation Range.*—When 100 ml. of the toluole are tested by the standard method, the difference between the temperatures (running-points) at which 5 ml. and 95 ml. of distillate have been collected shall not exceed 1·0 centigrade degree. The boiling-point shall be approximately 110° C.

5. *Acid Washing.*—When the toluole is tested by the standard method, the acid layer which separates shall be clear, and shall not be darker than a solution of 1·0 gm. of pure potassium dichromate in 1000 ml. of a mixture of equal volumes of pure sulphuric acid (containing 98 per cent. by weight of H_2SO_4) and distilled water.

6. *Total Sulphur.*—The toluole shall not contain more than 0·2 per cent. by weight of total sulphur.

7. *Freedom from Acids, Alkalies and Sulphuretted Hydrogen.* —The toluole shall not give a positive reaction for acids, alkalies or sulphuretted hydrogen.

8. *Residue on Evaporation.*—The toluole shall not leave more than 0·01 per cent. by weight of residue.

1929 Specification for Xylole

1. *Colour.*—The xylole shall not be darker than a freshly prepared solution of 0·2 ml. of 0·1 N. iodine solution in 1000 ml. of distilled water.

2. *Specific Gravity.*—The specific gravity of the xylole at 15·5° C./15·5° C. shall not be lower than 0·8600 nor higher than 0·8695.

3. *Water.*—The xylole shall be free from water and other visible impurities at 15·5° C.

4. *Distillation Range.*—When 100 ml. of the xylole are tested by the standard method, the difference between the temperatures (running-points) at which 5 ml. and 97 ml. of distillate have been collected shall not exceed 2·5 centigrade degrees. The boiling-point shall be approximately 140° C.

5. *Acid Washing.*—When the xylole is tested by the standard method, the acid layer which separates shall be clear, and shall not be darker than a solution of 2·0 gms. of pure potassium dichromate in 1000 ml. of a mixture of equal volumes of pure sulphuric acid (containing 98 per cent. by weight of H_2SO_4) and distilled water.

6. *Freedom from Acids, Alkalies and Sulphuretted Hydrogen.* —The xylole shall not give a positive reaction for acids, alkalies or sulphuretted hydrogen.

7. *Residue on Evaporation.*—The xylole shall not leave more than 0·01 per cent. by weight of residue.

8. *Paraffins.*—The xylole shall not contain more than 4·0 per cent. by volume of paraffins.

CHAPTER X

STEAM-RAISING

Plant.—A sufficiency of steam is an essential in a coke-oven and by-products plant. One of the best types used is the Babcock and Wilcox water tube boiler. These boilers are usually built of 4-inch tubes expanded into inclined headers which communicate with overhead drums and a small transverse mud drum in the rear. The overhead drums are most commonly longitudinal, 24 to 42 inches in diameter. The tubes incline downwards from front to rear, the header plane at right angles with the tubes. Steel forgings are used for pressures over 160 lbs. In the headers, opposite each tube end, is a hand hole with inside plate and ball-head bolt. The hand hole joint is either ground or provided with a thin gasket. The gases strike the tube banks transversely and usually cross them three times. Heat extraction is apt to be most thorough when the tube bank is higher than it is wide. This means high efficiency, a narrow boiler, and low first cost of installation, but may give an insufficiently wide grate for some fuels. Coke breeze may be successfully burnt under these boilers, and this can be economically handled by underfeed grate stokers.

When the economics of steam-raising is being considered, the following points must be taken into account :—

(1) Management.
(2) Capital cost.
(3) Fuel cost and method of delivery.
(4) Ash disposal.

(5) Water—condition and supply.

(6) Repairs and maintenance.

(7) Labour costs of operation.

Under boiler management come a great number of points. Time spent in the proper care of a boiler plant is of great importance. It should be regularly cleaned and inspected. All fittings should be occasionally examined to see that they are kept in proper working order. The condition of the boiler water is the first consideration, and the feed-water should be regularly tested for hardness and impurities. If the feed-water contains sediment or soluble salts, the boilers must be blown down at regular intervals. If scale is formed, or general corrosion in the form of pitting, the cause is the chemical action of impure feed-water. If the water is acid, it should be neutralised by some alkaline substance, such as carbonate of soda, before it enters the boilers. Soda should be used in the smallest possible quantity, as this substance may produce priming in the boilers.

The feed-water should be preheated, as certain solids may be precipitated by the rise in temperature. Some of the precipitates remain soft and can be blown out, while others deposit as hard scale. A thin coating of scale has been said to be beneficial as it keeps the water from direct contact with the iron, and prevents corrosion and pitting ; but the danger is that if a thin scale forms, a thicker one will follow, and this heavy scale, being a poor conductor of heat, not only causes waste of fuel, but allows overheating. Salts of lime (calcium), together with carbonate of magnesia, are the solids most frequently found in the water, and are the most troublesome. Hard water contains considerable quantities of lime. So-called soft water has usually little solid in suspension, but may contain impurities that cause corrosion or pitting.

Incrustation may be prevented by precipitating the scale-

forming substances before the feed-water reaches the boilers, by the introduction of chemical compounds to neutralise their evil effects or by removing the sediment before it becomes hard. There are many devices for the removal of scale by mechanical means. The tubes of a water tube boiler can be cleaned by an air or a water driven turbine, which rotates and swings cutters against the scale on the interior surface of the tube.

Boilers may be heated by different fuels, *e.g.* coal, small coke breeze, surplus gas or tar.

The Babcock and Wilcox type of underfeed travelling grate stokers consist of an endless chain of short grate bars or links moving over sprocket wheels at the front and rear end. These sprocket wheels are driven by a mechanism consisting of a gear train actuated by pawls and a ratchet, the arms carrying the latter being given a reciprocating motion by a rod and eccentric on a shaft. This shaft may be operated by any type of motor or engine, and the speed of the grate is regulated by varying the stroke of the arm carrying the pawls. The fuel is fed to a travelling grate through a hopper, extending the full width of the grate and mounted on the front of the frame. The depth of the fuel layer is regulated by a gate in the hopper. The coal or fuel is ignited at the front end and carried slowly to the rear end, the speed and thickness of the fire being so regulated that the fuel shall be completely burnt by the time it reaches the back, nothing but ashes being discharged into the pit. A flat firebrick arch in the front end aids combustion by igniting the fresh fuel as it enters and keeping up the temperature of the burning volatile gases.

It is essential that just sufficient air should enter the furnace to burn the fuel completely, and any excess only lowers the temperature. The rate at which the fuel burns is dependent upon the amount of air passing through the fuel bed, other conditions remaining the same. The difference between the draught above and below the fuel bed,

combined with the fuel bed resistance, fixes the rate at which the fuel will liberate gases, the furnace temperature remaining the same. Therefore, a uniform rate of fuel feed, and uniformity of refuse removal, afford the best opportunity for completion of combustion with a minimum air supply. It is always advisable to have a slight excess of air over the theoretical amount. Smoke coming from the boiler chimney signifies that there is insufficient air for proper combustion. All underfeed stokers depend upon forced draught for air supply through the fuel bed, and upon natural draught to carry the burning gases through the boiler setting.

Boiler Horse-power Unit.—One boiler horse-power is the evaporation of 30 lbs. of water per hour, from a temperature of 100° F. into steam at 70 lbs. gauge pressure. This is practically equivalent to the evaporation of $34\frac{1}{2}$ lbs. per hour from and at 212° F.

Efficiency of a Boiler is the weight of water evaporated per lb. of coal burnt. In comparing the evaporating powers of boilers, it is usual to find the actual number of heat units required to evaporate 1 lb. of the water at feed temperature into steam at the given pressure, and then divide the result by 996, which is the number of heat units required to evaporate 1 lb. of water from a feed temperature of 212° F. to steam at 212° F. This is called the *equivalent evaporation* from and at 212° F.

Example.—A boiler evaporates 7 lbs. of water per lb. of coal when working at 100 lbs. pressure absolute, the feed temperature being 60° F. Find the equivalent evaporation from and at 212° F.

Equivalent weight of water per lb. of coal

$$= \frac{w\{H - (t - 32)\}}{996},$$

where H is total heat of steam per lb. at boiler pressure from tables ;

t is actual temperature of feed-water ;

w is actual weight of water evaporated per lb. of coal.

Therefore,

$$\text{equivalent evaporation} = \frac{7\{1182 - (60 - 32)\}}{996}$$
$$= 8 \cdot 1 \text{ lbs.}$$

In first-class practice boilers may be expected to evaporate 10 lbs. of water per lb. of coal (from and at 212° F.) but frequently the figure is 7 lbs. or less.

Power of Boilers is usually measured by the quantity of dry steam in lbs. which can be generated per hour, or, in other words, by evaporative capacity, this being measured when the boiler is operating under normal working conditions. Thus, boilers rated to work at an evaporation of, say, 5000 lbs. per hour are capable of an increase of 25 per cent. above working load under forced draught for a short period. If a boiler of 5346 lbs. of steam per hour supplies an engine requiring 15 lbs. of steam per I.H.P. per hour, then the power obtained from that boiler is

$$\frac{5346}{15} = 356 \cdot 4 \text{ H.P.}$$

The Factories Act of 1937 requires that every steam boiler and all its fittings and attachments must be thoroughly examined by a competent person (a) at least once every 14 months and (b) after any extensive repairs. Such examination must be carried out (a) when the boiler is cold and the interior and exterior prepared in the prescribed manner and (b) when the boiler is under normal steam pressure. A signed report of such examination must be entered on the general register and, when the examiner is

a boiler inspector, the report must be countersigned by the engineer or other responsible person.

The Collin Process of Dry Cooling Coke.—The hot coke is discharged into cooling pockets, constructed below the flat coke bench (Fig. 16). For every three ovens one cooling pocket is arranged which is large enough to contain the coke from one oven. The coke is cooled in about 6
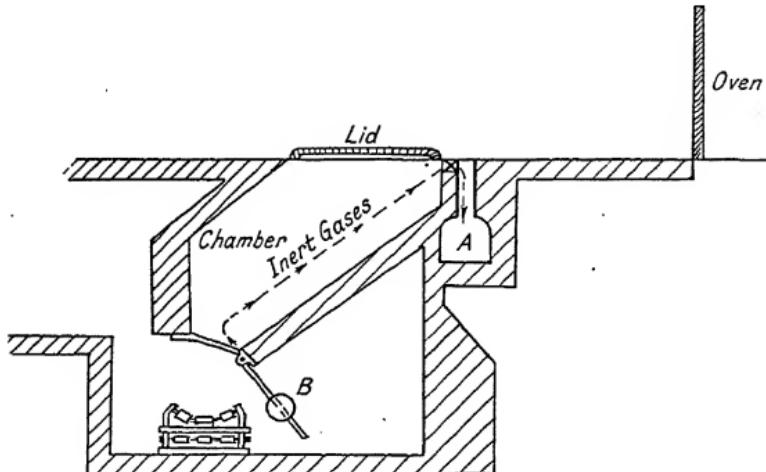
FIG. 16.—Collin Process of Dry Cooling Coke.

hours. Thus the pocket can easily be emptied by the time the neighbouring oven is ready to be discharged. The cooling pockets are closed at the bottom by a water cooled door, and at the top by a large cast iron door or lid. Each coke pocket is connected to the waste flue A, and the return main B. A suitable valve is arranged on each connecting flue, so that each pocket can be connected to or disconnected from circulation. At the end of the return main B, an electrically operated fan circulates inert gases through the hot coke and thus through flue A, to a tubular boiler plant, where the heat in the gases is taken up.

The cooled gases are then recirculated, the gases leaving the cooling chamber at approximately 700° C. and being cooled to about 200° C. in the boilers. The inert gases are passed through the hot coke only long enough for the coke to cool down to the desired temperature, when the gas inlet and outlet valves are closed. As ovens are continuously being discharged along the whole length of the battery there are always several pockets filled with hot coke, so that there is a continuous stream of hot gases to the boiler plant. During the cooling the pocket should be kept under a slight pressure so that no air can mix with the inert gases. The cooled coke is discharged through the bottom doors, and can be loaded directly into trucks or on to conveyors to the screening plant. It has been said that the cost of a Collin plant to operate 375 tons of coke daily is approx. £12,000 and that from 900 to 1000 lbs. of steam per ton of coke are thus raised.

Laboratory Tests of Boiler Water

The total hardness of both treated and untreated water is estimated by the addition of soap solution of known strength and is due to the fact that when a solution of soap is mixed with a solution of a calcium or a magnesium salt, an insoluble calcium or magnesium soap is produced. No lather will form, therefore, when water containing such salts is shaken with soap solution, until excess soap is present. This is indicated by the formation of a permanent lather, the latter being defined as a lather which remains stable, no bubble bursting, when the bottle is laid on its side for 5 minutes. The solution of soap, in methylated spirit and distilled water, is first standardised by titration against a standard solution of calcium chloride, and is then diluted to a convenient strength. The water is tested with this standard soap solution. The result should be given in English degrees of hardness, wherever the water consump-

tion is calculated in gallons, thus facilitating calculations
with respect to the softening medium required.

The English Degree of Hardness.—By definition, one
grain of chalk dissolved in one gallon of water confers on
that water a hardness of 1° and an alkalinity of 1°. These
degrees represent parts of calcium carbonate per 70,000 parts
of water (1 gallon = 70,000 grains). The strength of the
soap solution is such that each c.c. corresponds to 0·001
gram of calcium carbonate. In the actual test, 70 c.c. of
water are taken, so that if this quantity requires 1 c.c. of
soap solution it corresponds to 1 part in 70,000 or 1° hardness.

Solutions Required.—(a) *Water of Standard Hardness.*—
0·2 gram of pure Iceland spar is dissolved in a little hydro-
chloric acid, the beaker being covered with a watch-glass
to prevent loss by spirting. The solution is slowly evapor-
ated to dryness on the water bath or hot plate ; water is
now added and the solution again taken to dryness. This
is repeated three or four times to ensure the removal of all
free hydrochloric acid. The residue of neutral calcium
chloride is dissolved in distilled water and made up to one
litre.

(b) *Soap Solution.*—Thirty-two grams of pure, powdered
Castile soap are dissolved in 1000 c.c. of industrial methylated
spirit and 2000 c.c. of distilled water are added. This is
allowed to stand for at least 24 hours to ensure complete
solution of the soap.

Note.—For this purpose *industrial* methylated spirit must
be used. *Mineralised* methylated spirit cannot be employed
as it contains resins, petroleum oil and pyridine, in addition
to the aniline dye.

Standardising the Soap Solution.—Before using the
soap solution, it must be standardised against the standard

hardness water. This is accomplished by adding the soap solution 1 c.c. at a time, shaking vigorously after each addition, to 50 c.c. of standard hardness water. The latter is made up to 70 c.c. with distilled water. On nearing the end-point about 0·2 c.c. should be added at a time until a permanent lather is obtained. The soap solution should be adjusted so that 11 c.c. (1 c.c. for hardness of distilled water) just form a permanent lather with 50 c.c. of the standard hardness water. 1 c.c. of soap solution then corresponds to 0·001 gram of calcium carbonate, or a direct reading is obtained when titrated against 70 c.c. of the sample.

Total Hardness.—Measure out 70 c.c. of the water to be tested by means of a measuring cylinder. Run in the soap solution, 1 c.c. at a time, and proceed as above. The number of c.c. of soap solution used as indicated by the burette gives the total hardness. *E.g.* 70 c.c. of water require 20 c.c. of soap solution to give a permanent lather. As 70 c.c. of distilled water require 1 c.c. of soap solution to form a lather, the usual practice is to deduct one from the burette reading in order to arrive at the total hardness as found by the soap test; thus the above water has a hardness of 20 − 1 = 19°.

Classification.—Water having a total hardness of less than 8° (English) is considered soft, between 8° and 16° medium, and above 16° hard. If the water requires more than 16° of soap solution it must be diluted with one or more volumes of distilled water and the value found multiplied by the appropriate factor.

Alkalinity.—The total hardness of the water may be made up of (1) *temporary hardness,* due to the bicarbonates of calcium and magnesium ; or (2) *permanent hardness,* due to such salts as the sulphates and chlorides of calcium and

magnesium. Temporary hardness is usually removed by the addition of lime, when the insoluble normal carbonate is precipitated ; while permanently hard water is treated by the addition of soda-ash, when the insoluble carbonates are again formed, and the soluble sodium chloride and sulphate pass forward to the boiler. The nature of the hardness must, therefore, be determined before softening of the water can be successfully carried out. The temporary hardness is determined by titrating with 0·02 N. sulphuric acid using methyl orange as indicator. This gives the hardness due to bicarbonates, and the difference between this figure and the total hardness represents the permanent hardness.

Method of Determining Alkalinity of Untreated Water.— Measure out 70 c.c. of the water and pour into a beaker. Add 2 drops of methyl orange and run in 0·02 N. sulphuric acid until an orange-pink colour appears. The number of c.c. of acid used gives the temporary hardness. Should this alkalinity *exceed* the hardness as determined by the soap, then the water contains much carbonate of soda and there is no permanent hardness.

Method of Determining Alkalinity of Treated Water.— Measure out 70 c.c. as before and pour into beaker. Add 5 drops of phenolphthalein as indicator, when the sample will turn a .pink colour. Run in 0·02 N. sulphuric acid until the pink colour is discharged. Take the reading on the burette and add one or two drops of methyl orange ; continue the titration until the orange-pink colour appears. The results obtained should be recorded in the log book. The phenolphthalein figure gives the caustic alkalinity plus half the alkalinity due to carbonate of soda ; whereas the final figure obtained by the addition of acid gives the total alkalinity due to the caustic soda plus the carbonate. If, therefore, the final figure is less than twice the first figure, there are both caustic soda and carbonate present in the water.

Boiler Water.—The dangers of the impurities in the water in a boiler becoming very concentrated are apparent. Thus priming and bumping may result, together with corrosion of fittings, valves, etc. On the other hand, the alkalinity, which increases with concentration, must not be allowed to get below a fixed value, otherwise a slight fluctuation in the feed-water would be dangerous. A simple and straightforward test of the concentration of the water actually drawn from the drum is to take the specific gravity of the sample by means of a No. 1 Twaddell hydrometer. This figure should not exceed 3 ; should it do so, the boiler should be sludged more frequently. For the information of the laboratory, the alkalinity is determined as for the treated feed-water ; but decinormal sulphuric acid is usually employed, and the figure obtained is multiplied by 5.

CHAPTER XI

THE DESIGN AND OPERATION OF PLANT

Design and Layout

THE primary point in the location and design of plant is minimum cost taken in its broadest sense. The plant should be one which will give the best possible performance and the most remunerative return for the capital outlay.

Firstly, it is essential that the best type of oven is built to suit the coals which will be available. This includes the best width and height to produce a coke of the best mechanical strength. To find this out, it is advisable to get a quantity of the coal coked by some firm owning coke-ovens, and to carry out the usual shatter and Trommel tests, with laboratory analyses especially of ash and sulphur contents. A firm of contractors building ovens can give useful information, and can arrange for actual ovens, of the required design, to be seen in operation.

The next point.is to decide where to build the ovens ; the nearness of blast-furnace or coal-mine may determine this, but the actual site must then be carefully examined, particularly to ascertain that there is a good foundation. There should not be any old mine-workings underneath, nor should it be made-up ground. Bore-holes should be put down, and one or two trial holes dug down to firm foundations, before finally deciding on the site, to ensure that there is no risk of subsidence. The plant should be close to a railway, so that sidings into the plant can be added economically. The nearness of a road should be considered, to enable motor traffic, lorries, etc., to enter.

The railway sidings should be arranged to minimise shunting, and the line bringing in the coal trucks should slope down to the screening plant, so that these railway trucks, having been emptied of coal, may run by gravity to the coke filling station.

The ovens should be facing such a way that the coke is pushed out on the opposite side to where the by-products plant is situated. A railway siding should run alongside the storehouse, the fitting shop and the sulphate and exhauster houses, finally to the benzole filling point, which should be situated away from shunting operations. It is often advantageous to be near a river, from where abundant water for cooling may be procured.

A roadway should come into the plant for convenience of road transport, lorries, etc. ; this should run past the offices, the laboratory, the time office and men's meal and wash rooms, alongside the storehouse, the fitting shop and to the sulphate house (for local farmers to get bags of sulphate), and to the benzole drum filling platform.

In addition to a railway siding running along the other side of the fitting shop, it is useful to have a siding running into the shop itself. The plant as far as possible should be above-ground, with storage tanks in concrete enclosures which are easily drained of water. All pipes should as far as possible be above or on the ground, visible and easy to see, for repair purposes. Should they be underground, they should be in concrete open trenches which drain to a sump. All storage tanks should have large man-holes at ground level to facilitate cleaning.

The exhausters, pumps, compressors and sulphate centrifugals can all be housed in the one building, the sulphate store being next door. The benzole house should be a separate building apart from the others. Tiling of walls and floors, with floors sloped properly for drainage to ensure cleanliness, is good economy.

The cooling or condensing plant is best if 25 per cent.
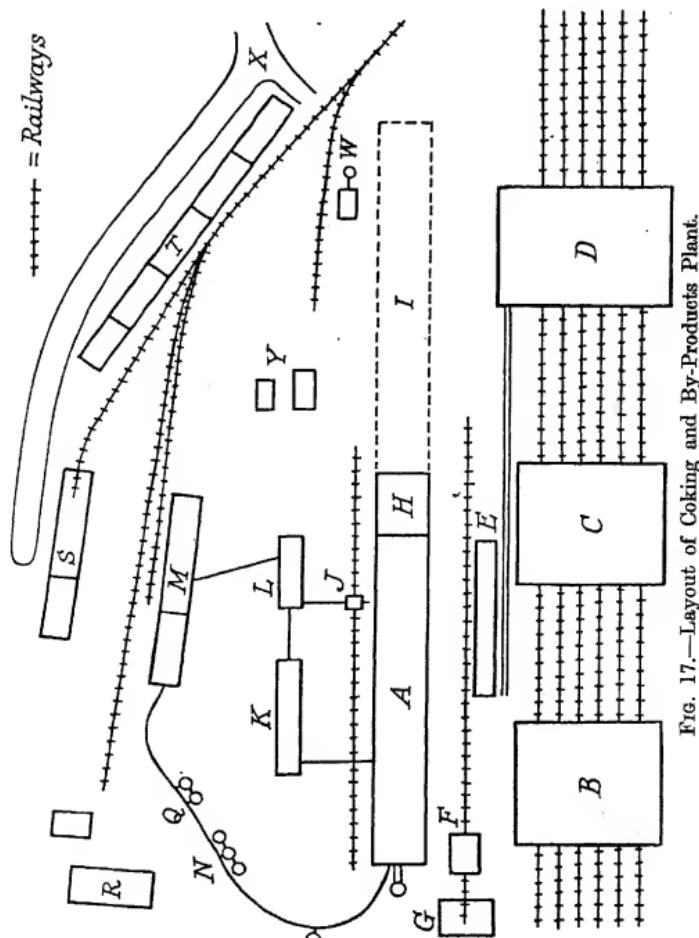
Fig. 17.—Layout of Coking and By-Products Plant.

A. Ovens.
B. Coal bunker.
C. Mixing bunker.
D. Coke screens.
E. Coke bench.
F. Coke car.
G. Quenching station.
H. Service bunker.
I. For extensions.
J. Ram or pusher.
K. Gas coolers.
L. Tar precipitators.
M. Sulphate and ex-
     hauster houses.
N. Benzole scrubbers.
Q. Gas coolers.
R. Benzole house.
S. Stores and fitting shop.
T. Offices, laboratory and
     meal house.
W. Boilers.
X. Roadway.
Y. Tar and acid storage.

+++++++ = *Railways*

M

above margin. Further, should there be any possibility of extending and building more ovens at a future date, it is advisable to make arrangements to make the by-products plant sufficiently large so that no additional plant is needed. Should finance not allow this, the layout must be arranged to allow additions when required.

The drainage of a new plant must be considered ; sumps, settling ponds and weirs are often useful to catch any spills before they get down the drains and possibly to streams or rivers where pollution may take place, perhaps poison cattle drinking, or annoy some fishery board.

Fig. 17. shows a suggested plan for a coking and by-products plant, not in existence but of a composite nature from different existing plants.

Laboratory Control

On a modern coking plant it is essential that the management have an efficient chemist and staff, to carry out the necessary daily and weekly tests and to keep in close touch with the processes. On some plants the chemist is put in responsible control of the benzole plant. Daily tests should be carried out on the following :—

(1) *Coal* for moisture, ash and volatile matter ; swelling test and fusibility of the ash if needed.

(2) *Coke* for moisture and ash, and if required for sulphur and phosphorus.

(3) *Gas* after ammonia extraction for ammonia.

(4) *Gas* after benzole scrubbers for benzole.

(5) *Wash Oil* after still for stripping efficiency.

(6) *Wash Oil* before still—distillation test.

(7) *Crude Benzole*—note percentage distilling over at 120° and 160° C.

(8) *Rectified Spirit*—National Benzole specification.

(9) *Sulphate of Ammonia*—moisture, acidity and ammonia content ; waste liquor.

(10) *Tar* despatched—for water content.

(11) *Feed-water* to boilers—for hardness.

Further tests should be carried out as required. If gas is sold, a full gas analysis should be made daily or as needed. A weekly shatter test of the coke should be made, as well as a screening test of the coke, and of the coal after the crushers. If an ammonia still is in use, the waste liquor should be tested daily for ammonia, the water content from the benzole condensers should also be tested.

Temperatures.—On the ovens the " heater," using an optical pyrometer, should take the flue temperatures of (*a*) one series, *e.g.* 1–11–21–etc., and (*b*) one complete wall per shift at least, and record these temperatures in a book. In addition, the gas temperatures should be noted hourly (1) before and after the coolers, (2) before and after the benzole scrubbers. The oil temperatures should be taken (3) before the benzole crude still and (4) after the oil coolers. Other temperatures as required should be noted at regular intervals.

Coal.—*Moisture (Coal or Coke)*.—Weigh out about 2 grams (powdered to pass 72 mesh B.S.S.) in a shallow silica dish with a well fitting cover. Heat uncovered coal for one hour at just over 105° C., cool the covered dish in a desiccator charged with concentrated sulphuric acid, and weigh covered. The loss of weight gives the percentage of moisture.

Ash.—After moisture estimation the same sample may be taken. Weigh out 1 gram and put into a silica dish at approximately 1 cm. depth by 5 cms. diameter, heating slowly at first and finally at a temperature of 750° to 800° C. for one hour in an oxidising atmosphere in a muffle furnace, until weight is constant. Cool in a desiccator and weigh. To test the completeness of combustion, moisten the ash

with a few drops of alcohol; if black specks appear, burn off the alcohol carefully and re-ignite. The percentage weight of ash, as well as the colour, are to be given.

Volatile Matter.—Weigh out 1 to 5 grams of coal finely powdered to pass 72 mesh in a platinum or nickel crucible, covered with a well fitting lid—adding about 3 drops of benzene—place the covered crucible on a support of platinum wire and heat for 7 minutes at a temperature of 950° to 980° C.; allow to cool and weigh.

Loss in weight = V.M. + moisture, to be given as percentage of volatile matter of the dry coal.

Sulphur.—Weigh 1 gram of finely powdered coal in a porcelain dish or platinum crucible and mix intimately with 3 grams of Eschka mixture (composed of 2 parts by weight of pure calcined magnesia and 1 part by weight of pure anhydrous sodium carbonate—pure quicklime may be substituted for magnesium oxide if the latter is not available in pure form). Cover the contents of the crucible with 1 gram of Eschka mixture. At first heat gently to ensure the slow expulsion of the volatile matter, then gradually raise the temperature to a red heat; continue heating at over 800° C. until the black-grey particles have completely disappeared. During the final heating, and not before, the mass should be stirred with a stout platinum or nickel wire to promote oxidation.

Transfer the charge to a beaker, wash the crucible thoroughly with hot water and digest with approx. 10 c.c. of bromine water; add hydrochloric acid in sufficient excess to dissolve the solid matter, boil to expel the bromine, wash and filter. Cool, neutralise with ammonia solution, using methyl orange as indicator, and add 4 c.c. of concentrated hydrochloric acid. Heat to boiling and add a slight excess of a 10 per cent. solution of barium chloride. Stand in a warm place overnight. Filter and wash with hot water until the washings show no opalescence with silver nitrate solution. Dry and ignite; after oxidising with a drop of

nitric acid and a drop of sulphuric acid, evaporate, re-ignite and weigh. (Weight of sulphur = weight of barium sulphate × 0·1374.)

Nitrogen (Kjeldahl Method).—Weigh 1 gram of coal ground to pass 100 mesh and transfer to a hard-glass Kjeldahl flask. Add 30 c.c. of pure, concentrated sulphuric acid, 7–10 grams of anhydrous sodium or potassium sulphate and 0·8–1·0 gram of metallic mercury. Heat the mixture gently until the initial frothing has ceased, then boil until colourless (2–6 hours). Allow to cool sufficiently to prevent violent reaction, then add a few crystals of finely ground potassium permanganate to complete oxidation. Dilute the cold solution to about 200 c.c. with water, fit the flask with a tap or thistle funnel and an efficient splash head, and connect through a condenser to a bulbed tube dipping into 20 c.c. of decinormal sulphuric acid (Fig. 18). Add through the funnel 100 c.c. of a solution containing 400 grams of sodium hydroxide and 40 grams of sodium sulphide per litre and distil over 150 to 200 c.c. Titrate the excess of sulphuric acid with decinormal sodium hydroxide or potassium hydroxide solution, using methyl orange as indicator.

Coking Index of Coal.—Weigh the requisite quantities of finely powdered coal and sand to give *exactly* 25 grams of a mixture, having the two ingredients in the desired proportions, in a tared, stoppered, cylindrical weighing bottle. Mix thoroughly and transfer the mixture to a crucible, rotating the bottle continuously during the process to prevent separation of the constituents of the mixture. Place the charged, covered crucible on a platinum or nichrome wire triangle, so adjusted that the bottom of the crucible is at a distance of 1 cm. above the top of the Méker burner, heat the crucible in the flame for 7 minutes and allow to cool.

Place a smooth cork on the coked mass, lay on the top of the cork a tared shallow porcelain dish, bottom up, invert the whole arrangement and remove the crucible carefully. Place the coked mass on a level surface, where it can be

tested by lowering the 500-gram weight upon it gently, and weigh the powder which then remains in the porcelain dish. The *maximum ratio of sand to coal in the mixture* which gives a coherent mass capable of bearing a 500-gram weight, the proportion of loose powder being less than 5·0 per cent., is adopted as the coking index.

Fresh Wash Oil.—Distil 100 c.c. of the oil from a distilling flask through an air condenser. Note the percentages of water and oil distilling at 200°, 250° and 300° C. (corrected). Cool the 200–300° C. fraction, separate any naphthalene, dry press and weigh. Take the viscosity at 20° C. Take the specific gravity at 15·5° C.

Benzolised and Debenzolised Oils.—Take the specific gravity at 15·5° C. Distil 300 c.c. from a distilling flask through a water condenser. Note water coming over and percentage of oil at 200°, 250° and 300° C. Redistil the fraction up to 200° C., after separation of water, in a retort as for crude benzole, noting the percentage (of the whole 300 c.c.) distilling at 120° C. and 160° C.

Crude Benzole.—*Daily Test.*—100 c.c. are distilled from a retort through a condenser (thermometer ⅜ inch from the bottom of the retort). Note percentage distilling over at 120° C. and at 160° C. (corrected). The correction for temperatures is made by adding or subtracting the amount by which the boiling-point of distilled water exceeds or falls short of 100° C.

For Sale Test.—Distil 100 c.c. as above, but cool residue in retort, pour into a receiver and measure how much has been lost in distillation, adding this to the percentage at 120° and 160° C. Take the specific gravity by means of a hydrometer after cooling to 15·5° C.

Ammonia in Gas after Ammonia Extraction.—*Aspirator.*—Suck 150 litres of gas by means of an aspirator

from the gas main through a dry gas wash-bottle and then through one containing 40 c.c. of sulphuric acid (370 c.c. of concentrated acid in 2 litres of water) further diluted up to a convenient bulk for the bottle. Wash this into a flask, make alkaline with caustic soda and distil into 10 c.c. of 10/17 N. sulphuric acid. Titrate the excess acid as for sulphate.

Meter.—Suck 100 litres of gas by means of a stream injector and meter through two wash-bottles in series each containing 5 c.c. of 10/17 N. sulphuric acid. Wash this into a beaker and titrate as for sulphate.

Sulphate of Ammonia.—*Moisture Test.*—Weigh 25 grams of the sample in a porcelain dish, heat in a steam-oven for one to two hours at over 100° C., cool in a desic-cator and weigh : repeat until weight is constant. Give the percentage of moisture lost.

Acidity Test.—Weigh out 20 grams and dissolve in distilled water. Filter and make up with washings to about 250 c.c. Titrate with decinormal caustic soda to a full yellow colour with methyl orange. To calculate the acidity :

Number of c.c. of 0·1 N.-NaOH × 0·0245
            = Percentage of free acid calculated as H_2SO_4.

Ammonia or Nitrogen Test.—Weigh out 3·3 grams of the sample. Wash through a funnel into flask E (Fig. 18), attached to condenser G. Into flask C run 50 c.c. of normal acid. Place as in sketch so that the end of the tube dips below the acid. Measure out 55 c.c. of normal caustic soda solution and pour into flask E by means of tap-funnel F. Rinse the measure out with water into flask E. Light gas under flask E, and heat slowly to boiling, and continue until the liquid just covers the bottom of flask E. All the ammonia will then be distilled over through the condenser into the acid in C. Open the tap-funnel F, turn gas off and, when cool, detach the funnel from E. Wash the

condenser through with distilled water into C.   The outside
of the tube also should be washed.   Add a few drops of
methyl orange solution to the liquid in C.   Then titrate
with decinormal caustic soda.   Shake till the free acid is
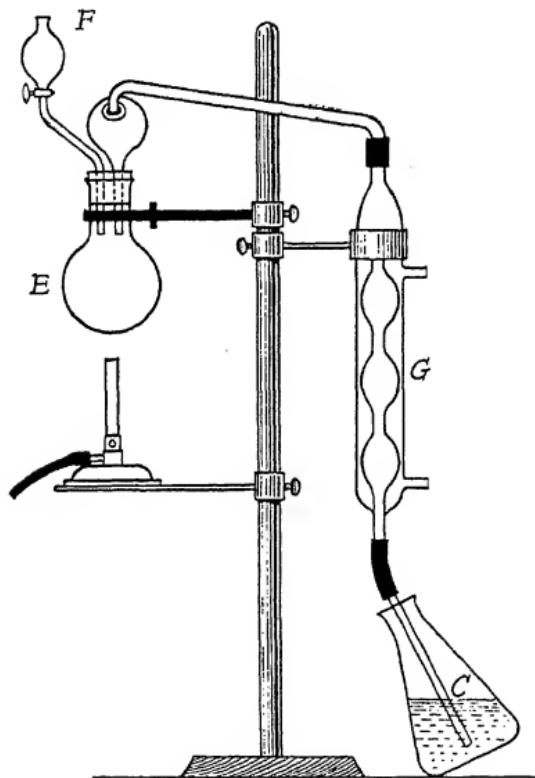
Fig. 18.—Laboratory Ammonia Test.

neutralised and the colour changes from orange to yellow.
Number of c.c. of normal acid neutralised by the ammonia

$$\times 0\cdot01401 = \text{grams of nitrogen};$$
$$\times 0\cdot01703 = \text{grams of ammonia}.$$

Give these in percentages.

Waste Liquor Test.—100 c.c. are made alkaline with caustic soda and distilled through a vertical condenser into 2 c.c. of 10/17 N. sulphuric acid, methyl orange being used as indicator. The excess acid is titrated against 10/17 N. sodium carbonate solution.

The Factories Act, 1937

It is essential that a coke-oven manager should have in his possession a copy of this act, and should make himself thoroughly acquainted with its sections. It came into force on 1st July, 1938. Of the 14 parts comprising this act, Part I covers the general principles of health, cleanliness and sanitation. The inside walls of workshops, meal-rooms, lavatories, etc. must be whitewashed or cleaned every 14 months. The District Council has certain powers for enforcing points on such matters.

Part II covers general provisions for safety ; this should be read carefully and it must be seen that every moving part is securely fenced in, and parts of the machinery made safe for the workmen. Every sump or pit must be covered or fenced. A table must be kept of all lifting tackle, etc., showing the safe working loads ; this must be posted in the store where the tackle is kept. All cranes and lifting machinery must be examined every 14 months. Suitable breathing and reviving apparatus must be kept and periodically inspected and a suitable number of persons must be practised in its use. There shall, as far as it is reasonably practicable, be provided and maintained safe means of access to every place at which any person has at any time to work ; when such a place is more than 10 feet high, secure hand-holds shall be provided, for safety. Where work has to be executed inside a tank or pit where dangerous fumes are liable to be present, special precautions must be taken ; such as to remove as far as is possible any fumes, and if some still remain, the person who enters shall wear

a rope of which the free end is held by some person outside, and the person entering shall wear a suitable breathing apparatus. No vessel which has contained an inflammable substance shall be welded or cut by the application of heat, until all steps have been taken to render it non-explosive and non-inflammable ; and after such operation no such inflammable substance shall be allowed to enter the tank until the metal has sufficiently cooled. Every steam boiler and all its fittings shall be examined by a competent person at least every 14 months, and a report of such an inspection inserted in the general register.

Regulations about steam receivers or containers include that a correct steam pressure gauge be fitted, which must indicate the pressure of steam, and except where only one steam receiver is in use, it shall bear a plate with a distinctive number easily visible. An examination of every steam receiver and all fittings must be carried out at least every 26 months, and the report entered in the general register. All air receivers shall have plainly marked on them the safe working pressure, and in cases where more than one receiver is in use, a number or distinguishing mark shall be clearly visible. Every air receiver shall be recorded in the general register.

Gas holders must be properly maintained, and shall be thoroughly examined externally once every two years, and such recorded in the register. If any lift of the gas holder has been in use more than 20 years, the internal state of the sheeting shall within two years, and thereafter at least once per ten years, be examined by cutting samples from the crown and sides of the holder, and such examination signed by the person making it for future inspection.

Where more than 20 persons are employed in one building, such a factory shall be certified by the District Council as being provided with such means of escape in case of fire as may be reasonably required. It shall be the duty of the

Council to examine every factory and if satisfied to give such a certificate.

There are new provisions as to welfare. An adequate supply of wholesome *drinking water* shall be provided, and suitable drinking vessels or an upward jet provided for drinking purposes. Also shall be provided and maintained for the use of employed persons *adequate and suitable washing facilities*, including soap and clean towel, or other suitable means of cleaning and drying. Thus a suitable wash room must be provided for the workmen, also adequate and suitable accommodation for clothing not worn during work hours, and arrangements for drying such clothing. A *first-aid* box or cupboard of the prescribed standard must be provided and maintained ; each box shall be in the charge of a responsible man. Where more than 50 men are employed, as on most coking plants, some man or men must be trained in first-aid treatment, and a notice shall be put up stating the name of the responsible person in charge of the first-aid box or cupboard.

Part IV deals with the hours and employment of young persons, such as apprentices, who must not work more than nine hours daily, nor more than 48 hours in any week. This refers to those under 16, and stipulates meal times, rest, etc. A notice shall be posted up stating :—the period of work each day, and the intervals allowed for meals or rest to such young persons. Young persons within the meaning of the act must not work on Sundays or Bank Holidays. Such young persons must be examined by a physician, who shall give a certificate stating whether he is fit for such employment.

This act applies to persons employed in connection with electricity or anything relating to the control of electrical energy.

The coke-oven manager must post up in a prominent position the following :—

Form 395 : Official Cautionary Notice under the Chemical

Regulations which includes use of Breathing Apparatus and Artificial Respiration.

Form 984 : Chemical Works Regulations 1922.

Form 1 :   Abstract of Factories Act 1937, with Summary of Workmen's Compensation Act 1925.

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An asterisk (*) indicates that a subject is illustrated.